No. 2016-2565

IN THE

United States Court of Appeals

FOR THE FEDERAL CIRCUIT

SNF HOLDING COMPANY, FLOPAM INC., CHEMTALL, INC., SNF SAS, SNF (CHINA) FLOCCULANT CO. LTD.,

Appellants,

v.

BASF CORPORATION,

Appellee.

APPEAL FROM THE UNITED STATES PATENT AND TRADEMARK OFFICE, PATENT TRIAL AND APPEAL BOARD IN NO. IPR2015-00600

PETITION FOR REHEARING AND REHEARING EN BANC

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Dated: November 16, 2017

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FORM 9. Certificate of Interest

Form 9
Rev. 10/17

UNITED STATES COURT OF APPEALS FOR THE FEDERAL CIRCUIT

SNF Holding Company, Flopam Inc., Chemtall Incorporated, SNF SAS, SNF (China) Flocculent Co. Ltd **v.** BASF Corporation

Case No. 16-2565

CERTIFICATE OF INTEREST

Counsel for the:
■ (petitioner) ☐ (appellant) ☐ (respondent) ☐ (appellee) ☐ (amicus) ☐ (name of party)

certifies the following (use "None" if applicable; use extra sheets if necessary):

1. Full Name of Party Represented by me	2. Name of Real Party in interest (Please only include any real party in interest NOT identified in Question 3) represented by me is:	3. Parent corporations and publicly held companies that own 10% or more of stock in the party
SNF Holding Company	SNF Holding Company	SPCM SA (France), which is the direct parent company (directly or indirectly) of all appellants.
Flopam Inc.	Flopam Inc.	
Chemtall Incorporated	Chemtall Incorporated	
SNF SAS	SNF SAS	
SNF (China) Flocculant Co. Ltd.	SNF (China) Flocculant Co. Ltd.	

4. The names of all law firms and the partners or associates that appeared for the party or amicus now represented by me in the trial court or agency or are expected to appear in this court **(and who have not or will not enter an appearance in this case)** are:

See attached.

FORM 9. Certificate of Interest **Form 9**
Rev. 10/17

5. The title and number of any case known to counsel to be pending in this or any other court or agency that will directly affect or be directly affected by this court's decision in the pending appeal. *See* Fed. Cir. R. 47. 4(a)(5) and 47.5(b). (The parties should attach continuation pages as necessary).

BASF Corp. v. SNF Holding Co., No. 4:14-cv-02733 (S.D. Tex.)

November 16, 2017
Date

/s/ James W. Dabney
Signature of counsel

Please Note: All questions must be answered

James W. Dabney
Printed name of counsel

cc: Counsel of Record

Reset Fields

The names of all law firms and the partners or associates that appeared for the party or amicus now represented by me in the trial court or agency or are expected to appear in this court (and who have not or will not enter an appearance in this case) are:

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STATEMENT OF COUNSEL
PURSUANT TO FEDERAL CIRCUIT RULE 35(b)(2)

Based on my professional judgment, I believe this appeal requires an answer to one or more precedent-setting questions of exceptional importance:

1. Whether 35 U.S.C. § 144 requires this Court to issue an opinion when exercising original jurisdiction under 35 U.S.C. §§ 141–144.

2. Whether 35 U.S.C. § 316 authorizes the United States Patent and Trademark Office ("PTO") to change an instituted ground of *inter partes* review ("IPR"), post-institution, in a manner that conflicts with the ground as stated in the petition on which IPR was instituted.

3. Whether a patentee, in an instituted IPR, can impeach its own admissions in the specification of a challenged patent.

November 16, 2017

/S/JAMES W. DABNEY
James W. Dabney
Counsel for Appellants

POINTS OF LAW OR FACT
OVERLOOKED OR MISAPPREHENDED BY THE PANEL

Pursuant to Federal Circuit Rules 35(e)(3)(F) and 40(a)(4), Petitioners provide this statement of points of law or fact that were overlooked or misapprehended by the panel:

1. The panel overlooked that, when it reviews final written decisions in IPR proceedings, this Court exercises a form of original jurisdiction and is required by statute to issue "its mandate and opinion, which shall be entered of record in the Patent and Trademark Office and shall govern the further proceedings in the case," 35 U.S.C. § 144, just as Federal Rule of Civil Procedure 52(a) requires district courts to state their conclusions of law when exercising original jurisdiction without a jury.

2. The panel overlooked that an IPR petitioner is master to decide "the grounds on which the challenge to each claim is based." 35 U.S.C. § 312(a)(3). In this case, a Patent Trial and Appeal Board ("Board") changed an already-instituted ground of IPR to have substantially different requirements than the Petition and the Board's institution decision had identified. The Board's action violated Petitioner's rights under 35 U.S.C. § 312(a)(3) and 5 U.S.C. § 554.

3. The panel overlooked that an instituted "ground" of IPR, within the meaning of 35 U.S.C. § 312(a)(3), includes a challenged patent's specification description of preexisting knowledge and skill in the art. "Admissions in the specification

regarding the prior art are binding on the patentee for purposes of a later inquiry into obviousness." *PharmaStem Therapeutics, Inc. v. ViaCell, Inc.*, 491 F.3d 1342, 1362 (Fed. Cir. 2007). A patentee, in an instituted IPR, cannot rightly impeach its own admissions in the specification of a challenged patent.

BACKGROUND

Respondent is the owner of U.S. Patent No. 5,633,329 (the "'329 Patent"; Appx25-33). The '329 Patent discloses and claims a process for preparing high molecular weight polymers that comprises making *hydrous*, rather than *anhydrous*, polymer gel in a tubular reactor having a conical bottom taper, and "removing the gelatinous reaction mixture by injection of an inert gas." Appx33 at col. 14, lines 39–40.

The Board's Institution Decision

Petitioners sought *inter partes* review of the '329 Patent on the basis of prior art showing (i) making hydrous polymer gel in a tubular reactor having a conical bottom taper, and (ii) removing the hydrous gel by injection of an inert gas. After receiving a preliminary response from the patentee, the Board instituted IPR as to all claims of the '329 Patent. Appx178-195.

In its institution decision, the Board construed the '329 Patent claim phrase, "removing the gelatinous reaction mixture by injection of an inert gas" (Appx33 at col. 14, lines 39–40), as describing the polymer gel "removing" operation that was disclosed in U.S. Patent No. 3,784,597 (the "'597 Patent" Appx499-502). The Board stated that "the '597 patent discloses *successfully removing* polymer gels from a tubular reactor" and cited column 3, lines 70–73 of the '597 Patent as "disclosing *the successful removal* of polyacrylamide gels from a tubular reaction vessel using inert gas pressure." Appx189 (emphasis added). The Board also stated, "Patent Owner has not explained why the challenged claims, as properly construed, require any particular efficiency level for the removal of the polymer gel, much less 'complete removal' of the gel from the reaction vessel." Appx190.

The Patentee Repudiates the '329 Patent's Background Disclosure

In its response to the Petition, the patentee asserted, "prior to the invention of the '329 patent, *conical tapers and inert gas had never been used* to remove polymer gels from reactors." Appx258 (emphasis added). In support of this assertion, the patentee submitted a declaration of one F. Joseph Schork who broadly asserted: "The prior art reflects that *those who attempted to use inert gas to remove polymer gel from a reactor found that it was not a viable option*." Appx2651 (emphasis added).

These statements directly contradicted the specification of the '329 Patent, which stated that the patentee had itself made polymer gel in a tubular reactor having a conical bottom taper, "the plastic polymer matrix . . . being discharged from the tubular reactor virtually without residue by injecting an inert gas." Appx27 at col. 2 lines 1–4, describing a process disclosed in U.S. Patent No. 5,081,215 (the "'215 Patent")). The patentee's expert admitted that he did not review the '215 Patent, despite its being cited and its disclosed process described in the '329 Patent specification. Appx3317-3318. His assertion, "those who attempted to use inert gas to remove polymer gel from a reactor found that it was not a viable option" (Appx2651), purported to impeach the '329 Patent specification's own description of what was known in the art.[1]

The Board's Final Written Decision

In its final written decision, the Board adopted a substantially different construction of the '329 Patent than it had adopted in its institution decision. For the first time, the Board held that the '329 Patent claim phrase, "removing the gelatinous reaction mixture by injection of an inert gas," required injection of inert gas into a reactor vessel for a *sufficient length of time* that "substantially all the gelatinous reaction mixture is discharged from the reactor." Appx8. And based on this new construction, the Board reversed itself and held that the '597 Patent did not disclose "*removing* . . . by injection of an inert gas" because Petitioner's expert's initial testimony on that point had "presume[d] that 'discharging' is equivalent to

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[1] In its opening brief to this Court, the patentee asserted, "[w]hile the '215 patent is prior art under 35 U.S.C. § 102(e), it also is a BASF invention and therefore not citable for purposes of an obviousness inquiry according to 35 U.S.C. § 103(c)." Appx3884. Patentee has since conceded that its reliance on 35 U.S.C. § 103(c) was error (Appx3893); but by then the Board had already been misled into relying on the patentee's erroneous argument and false evidence.

'removing.'" Appx14. *Contra* Appx189 (stating in the institution decision that "the '597 patent discloses *successfully removing* polymer gels from a tubular reactor" (emphasis added)).

The Board appeared to note the contradiction between (i) the patentee's assertion that "conical tapers and inert gas had never been used to remove polymer gels from reactors" (Appx258), on the one hand; and (ii) the '329 Patent specification statement that the '215 Patent process yielded polymer gel "being discharged from the tubular reactor virtually without residue by injecting an inert gas," on the other. Appx27 at col. 2, lines 3–4. The Board attempted to reconcile this contradiction by noting that the '215 Patent disclosed using a tubular reactor in conjunction with a "discharge aid" and concluded: the "use of a discharge aid in the '215 patent reactor tends to support Patent Owner's argument that one of ordinary skill in the art would not have had a reasonable expectation of success in using a conical taper and inert gas pressure *alone* (*the combination proposed by Petitioner*) to remove a sticky, gelatinous product from a reactor." Appx19 (emphasis added).

In fact, the grounds on which Petitioner had sought, and the Board had instituted, IPR were not restricted to "using a conical taper and inert gas pressure *alone*." Appx19. That notion and the potential relevance of a "discharge aid" in the validity analysis were contrary to the ground on which IPR was instituted and were first identified, without prior notice, in the Board's final written decision.

This Court Issues a Judgment Without Opinion

Petitioners timely appealed the Board's decision to this Court under the provisions of 35 U.S.C. §§ 141–144 and 319. Following oral argument on October 10, 2017 (Appx3885-3902), this Court issued a judgment which affirmed the Board's decision without opinion.

ARGUMENT FOR PANEL AND EN BANC REHEARING

I. THE PANEL'S DECISION CONTRAVENES 35 U.S.C. § 144

35 U.S.C. § 144 provides:

> The United States Court of Appeals for the Federal Circuit shall review the decision from which an appeal is taken on the record before the Patent and Trademark Office. Upon its determination the court shall issue to the Director its mandate and opinion, which shall be entered of record in the Patent and Trademark Office and shall govern the further proceedings in the case.

On its face, 35 U.S.C. § 144 requires this Court to issue an "opinion" when it reviews a Board decision under 35 U.S.C. §§ 141–144 and 319. This requirement reflects that this Court exercises *original* jurisdiction when it reviews Board decisions under 35 U.S.C. §§ 141–144 and 319. *See, e.g.*, *Wagner v. FEC*, 717 F.3d 1007, 1013 (D.C. Cir. 2013) (former 2 U.S.C. § 437h granted circuit courts of appeals "exclusive original jurisdiction" to hear certain election law claims); *Am. Portland Cement All. v. EPA*, 101 F.3d 772, 775 (D.C. Cir. 1996) (former 42 U.S.C. § 7006 granted D.C. Circuit "original jurisdiction to review three specific types of agency action"); *see also Marbury v. Madison*, 5 U.S. (1 Cranch) 137, 175 (1803) (holding that a judicial action seeking review of an executive action is an exercise of "original" not "appellate" jurisdiction); 16B Charles A. Wright et al., *Federal Practice and Procedure* § 4005, at 149 (3d ed. 2012) ("It has been widely supposed that the first review of 'quasi-judicial' determinations by administrative agencies cannot be characterized as appellate."); Louis L. Jaffe, *Judicial Control of Administrative Action* 263 n.5 (1965) ("the first reviewing court is a court of 'original' jurisdiction").

The "opinion" requirement of 35 U.S.C. § 144 parallels the similar requirement that Federal Rule of Civil Procedure 52(a) imposes on district courts when they review agency actions under 35 U.S.C. § 145 or other applicable statutes providing for judicial review. Rule 52(a) provides that in an action tried without a jury, a district court must "state its conclusions of law" and these "may appear in an opinion or a memorandum of decision filed by the court." The plain meaning of "opinion" in Rule 52(a) and 35 U.S.C. § 144 is:

> The statement by a judge or court of the decision reached in regard to a caused tried or argued before them, expounding the law as applied to the case, and detailing the reasons upon which the judgment is based.
>
> An expression of the reasons why a certain decision (the judgment) was reached in a case. . . .

Opinion, *Black's Law Dictionary* 1092 (6th ed. 1990); *see also* Dennis Crouch, *Wrongly Affirmed Without Opinion*, 52 Wake Forest L. Rev. 561, 573 (2017).

The Court in this case issued a “Notice of Entry of Judgment Without Opinion.” Appx3903. While this practice may be permissible in appeals from district court judgments, *see* Fed. R. App. P. 36(a) (distinguishing between judgments issuing with and without opinions), the text of 35 U.S.C. § 144 leaves no room for doubt but that an “opinion” must be issued when this Court exercises original jurisdiction under 35 U.S.C. §§ 141–144 and 319. There are strong institutional reasons for the “opinion” requirement.

“Inter partes review . . . preserves the ‘complete authority’ of Article III courts ‘to insure the proper application of the law’ with respect to questions of patentability . . . .” Brief for the Federal Respondent at 35, Oil States Energy Servs., LLC v. Greene’s Energy Grp. LLC, No. 16-712 (U.S. Oct. 23, 2017), 2017 WL 4805230, at *35 (quoting *Crowell v. Benson*, 285 U.S. 22, 46–47 (1932)). Knowing the reason(s) why a patent claim survived an IPR is also crucial to proper administration of 35 U.S.C. § 315(e)(2), which requires inquiry into whether an invalidity defense is one that a petitioner “reasonably could have raised during that inter partes review.” Without an opinion, a judgment can be open to many different interpretations. *Cf. KSR Int’l Co. v. Teleflex Inc.*, 550 U.S. 398, 418 (2007) (“To facilitate review, this analysis should be made explicit.”).

The Board in this case repeatedly indicated that it was not considering certain evidence and arguments on procedural, non-merits grounds. *See, e.g.*, Appx16

("We decline to consider this testimony because Petitioner did not cite to it or explain its significance in the Reply."); Appx19 at n.6 ("Petitioner does not make this argument, and this is not the combination of prior art elements proposed in the Petition."). During oral argument, members of the panel voiced skepticism at the Board's distinction between "removing" and "discharging." *See, e.g.*, Appx3894 ("in the context of this patent, not particularly from the word 'removed,' which I must say is not to my mind very distinct from the word 'discharge'"). Without an opinion, it is impossible to know the actual basis of this Court's judgment.

The "opinion" requirement of 35 U.S.C. § 144 is also important to achieving "desirable uniformity" in patent law, *Markman v. Westview Instruments, Inc.*, 517 U.S. 370, 390 (1996), and to enforcing "public and private standards of equity," *Precision Instrument Mfg. Co. v. Auto. Maint. Mach. Co.*, 324 U.S. 806, 816 (1945). Judicial review of Board decisions requires the searching inquiry prescribed by 5 U.S.C. § 706. *See Dickinson v. Zurko*, 527 U.S. 150, 152 (1999). These safeguards are promised to IPR petitioners under 35 U.S.C. § 319 and they are not protected when this Court does not abide the "opinion" requirement of 35 U.S.C. § 144, just as district court review of agency action must include articulated "conclusions of law." Fed. R. Civ. P. 52(a).

II. 35 U.S.C. § 316 DOES NOT AUTHORIZE THE PTO TO CHANGE THE GROUND OF AN IPR, POST-INSTITUTION, IN A MANNER THAT CONTRAVENES THE PETITION'S STATED GROUND.

35 U.S.C. § 312(a)(3) provides that a Petition for IPR must specify "the *grounds* on which the challenge to each claim is based" (emphasis added), and 35 U.S.C. § 315(e)(2) provides that "[t]he petitioner in an inter partes review of a claim . . . that results in a final written decision . . . may not assert . . . that the claim is invalid on any *ground* that the petitioner raised or reasonably could have raised during that inter partes review" (emphasis added). The statutory scheme clearly entitles an IPR petitioner to know in advance, and to identify in a Petition for IPR, the specific grounds on which it seeks IPR and to have *those grounds* evaluated and determined by the Director of the PTO or his or her designee. The "grounds" in an IPR petition typically do, and in this case did, include a statement of position that challenged patent claims refer to certain structure, material, or acts.

Although the PTO is free to conclude that grounds stated in a Petition do not warrant institution of IPR, there is no statutory authorization for the PTO to institute IPR on grounds that are *different* from those presented in a Petition or to force a Petitioner to risk estoppel under 35 U.S.C. § 315(e)(2) based on an adjudication of *different* grounds of invalidity from those that were presented in a Petition; but even assuming that such authority existed, 5 U.S.C. § 554 would clearly require the agency to notify a Petitioner that *instituted grounds* of invalidity were *no longer* *operative* and to provide the Petitioner with an opportunity to respond to a proposed post-institution change of grounds. That was not done here.

"IPR proceedings are formal administrative adjudications subject to the procedural requirements of the Administrative Procedure Act ('APA')," and "an agency may not change theories in midstream without giving respondents reasonable notice of the change and the opportunity to present argument under the new theory." *SAS Inst., Inc. v. ComplementSoft, LLC.*, 825 F.3d 1341, 1351 (Fed. Cir. 2016), *cert. granted*, 137 S. Ct. 2160 (2017) (internal quotation marks omitted). *SAS* vacated a final Board decision which construed a patent claim phrase as having a different scope than the Board had construed the same phrase to have in its institution decision. *Id*. at 1351–52.

As noted above, the Board in this case *instituted* IPR on the basis that the claim phrase, "removing . . . by injection of an inert gas" described a removing *operation* that was disclosed in the '597 Patent; but in its final written decision, the Board took the position that the claim phrase "removing . . . by injection of an inert gas" described a *result*—"substantially all the gelatinous reaction mixture is discharged"—which the Petitioners purportedly had not shown was disclosed in the '597 Patent. Appx8. This new construction of the '329 Patent effectively changed the grounds on which the Board reviewed the challenged claims, contrary not just to the Petitioners' right to identify the grounds of an IPR under 35 U.S.C.

§ 312(a)(3), but also to the advocacy opportunities that 5 U.S.C. § 554 and 35 U.S.C. §§ 311–319 guarantee as a basis for taking the risk of the estoppels prescribed in 35 U.S.C. § 315(e).

35 U.S.C. § 316 is not rightly construed to authorize the PTO to change the substantive content of an instituted ground of IPR, post-institution; but if such authority is recognized, the petitioner should be held entitled to the full panoply of advocacy opportunities that 35 U.S.C. §§ 311–319 guarantee those who petition for IPR on a stated ground.

III. A PATENTEE, IN AN INSTITUTED IPR, CANNOT RIGHTLY IMPEACH ITS OWN ADMISSIONS IN THE CHALLENGED PATENT SPECIFICATION.

In framing their Petition in this case, the Petitioners could not reasonably have anticipated that the patentee would attempt to repudiate the ’329 Patent specification and assert, in direct contradiction of that specification, that “prior to the invention of the ’329 patent, *conical tapers and inert gas had never been used* to remove polymer gels from reactors.” Appx258 (emphasis added). It was not until the oral argument in this case (*see* Appx3893) that it emerged that the patentee’s false statements to the Board regarding the prior art were purportedly based on an erroneous belief that, “[w]hile the ’215 patent is prior art under 35 U.S.C. § 102(e), it also is a BASF invention and therefore not citable for purposes of an obviousness inquiry according to 35 U.S.C. § 103(c).” Appx3884. In fact, because the application for the ’329 Patent was filed prior to November 29, 1999, the ’215 Patent was unquestionably prior art to the ’329 Patent under 35 U.S.C. § 102(e) (1994), and it was also prior art under 35 U.S.C. § 102(b) (1994).

Just as the “grounds” (35 U.S.C. § 312(a)(3)) on which IPR was instituted in this case included the Petition’s statement of position with respect to what structure, material, and acts were encompassed by the challenged claims of the ’329 Patent, those “grounds” equally included the specification text of the ’329 Patent. That text, which described preexisting knowledge and skill in the art, should have been held “binding on the patentee for purposes of a later inquiry into obviousness.” *PharmaStem*, 491 F.3d at 1362; *accord Constant v. Advanced Micro-Devices, Inc.*, 848 F.2d 1560, 1570 (Fed. Cir. 1988) (“A statement in a patent that something is in the prior art is binding on the applicant and patentee for determinations of anticipation and obviousness.”).

It was only by withholding the ’215 Patent from its expert that the patentee could induce him to assert, falsely, that “[t]he prior art reflects that *those who attempted to use inert gas to remove polymer gel from a reactor found that it was not a viable option*.” Appx2651 (emphasis added). The Board was clearly misled by this inadmissible argument, concluding that “use of a discharge aid in the ’215 patent reactor tends to support Patent Owner’s argument that one of ordinary skill in the art would not have had a reasonable expectation of success in using a conical taper and inert gas pressure alone (the combination proposed by Petitioner) to remove a sticky, gelatinous product from a reactor." Appx19.

The constraints that 35 U.S.C. § 316 impose on petitioners in IPR proceedings make even more important enforcement of the principle that "[a]dmissions in the specification regarding the prior art are binding on the patentee for purposes of a later inquiry into obviousness." *PharmaStem*, 491 F.3d at 1362. The patentee here succeeded in persuading the Board to conclude that the skill level in the art of the '329 Patent did not include the skill required to achieve what the '329 Patent specification, on its face, stated had already been achieved, namely, "the plastic polymer matrix . . . being discharged from the tubular reactor virtually without residue by injecting an inert gas." Appx27 at col. 1, line 65–col. 2, line 4.

In practical effect, by permitting the patentee to impeach the '329 Patent specification in this fashion, the Board changed the grounds on which IPR had been instituted without statutory authorization, and exposed Petitioners to risk of estoppel under 35 U.S.C. § 315(e)(2) that their Petition did not authorize. Rehearing en banc is appropriate to establish that an instituted "ground[]" of IPR, within the meaning of 35 U.S.C. § 312, includes the challenged patent's specification description of preexisting knowledge and the level of skill in the art.

CONCLUSION

For the reasons set forth above, this petition for panel rehearing or, alternatively, for rehearing en banc, should be granted.

Dated: November 16, 2017

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ADDENDUM

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Trials@uspto.gov Paper 49
571-272-7822 Entered: August 2, 2016

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SNF HOLDING COMPANY,
FLOPAM INC.,
CHEMTALL INCORPORATED,
SNF SAS, and
SNF (CHINA) FLOCCULANT CO. LTD.,
Petitioner,

v.

BASF CORPORATION,
Patent Owner.

Case IPR2015-00600
Patent 5,633,329

Before GRACE KARAFFA OBERMANN, JON B. TORNQUIST, and JEFFREY W. ABRAHAM, *Administrative Patent Judges.*

TORNQUIST, *Administrative Patent Judge*.

FINAL WRITTEN DECISION
35 U.S.C. § 318(a) and 37 C.F.R. § 42.73

I. INTRODUCTION

SNF Holding Company, Flopam Inc., Chemtall Incorporated, SNF SAS, and SNF (China) Flocculant Co. Ltd. (collectively "Petitioner") filed a Petition (Paper 1) requesting institution of *inter partes* review of claims 1–7 of U.S. Patent No. 5,633,329 (Ex. 1001, "the '329 patent"). BASF Corporation ("Patent Owner") filed a Preliminary Response (Paper 8, "Prelim. Resp.") to the Petition. Upon consideration of the Petition, we determined that the information presented in the Petition demonstrated a reasonable likelihood that Petitioner would prevail with respect to claims 1–7 of the '329 patent. Paper 9 ("Inst. Dec." or "Institution Decision"). Thus, pursuant to 35 U.S.C. § 314(a), we instituted trial with respect to those claims. *Id.*

Following institution of trial, Patent Owner filed a Patent Owner Response (Paper 20, "PO Resp."), to which Petitioner filed a Reply (Paper 28, "Pet. Reply"). Patent Owner also filed a Motion to Exclude (Paper 37), to which Petitioner filed an Opposition (Paper 42), and Patent Owner filed a Reply (Paper 46).

Patent Owner also filed a paper identifying allegedly improper arguments and evidence submitted in Petitioner's Reply (Paper 31), to which Petitioner filed a response (Paper 32). Finally, Petitioner filed a motion to exclude certain errata sheets filed by Patent Owner (Paper 43), to which Patent Owner filed an opposition (Paper 45).

An oral hearing was held on May 5, 2016, and a transcript of the oral hearing is included in the record. Paper 48 ("Tr.").

We have jurisdiction under 35 U.S.C. § 6(c) and this Final Written Decision is entered pursuant to 35 U.S.C. § 318(a) and 37 C.F.R. § 42.73.

For the reasons set forth below, we conclude that Petitioner has not demonstrated, by a preponderance of the evidence, that claims 1–7 of the ’329 patent are unpatentable.

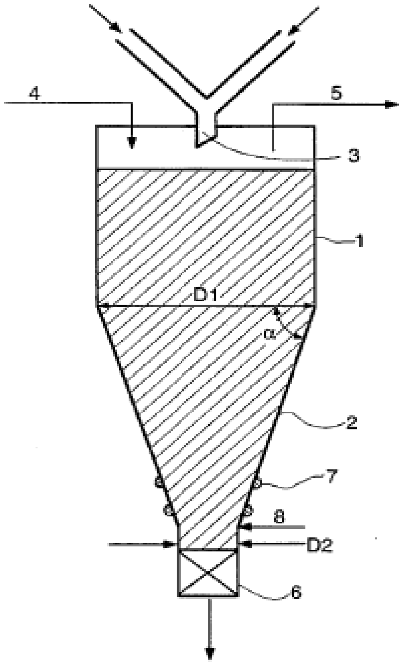
A. Related Matter

The parties indicate that the ’329 patent is being asserted in *BASF Corp. v. SNF Holding Co.*, No. 4:14-cv-02733 (S.D. Tex.), filed September 23, 2014. Pet. 1; Paper 6, 2.

B. The ’329 Patent

The ’329 patent discloses a process for polymerizing water-soluble, monoethylenically unsaturated monomers in an aqueous solution. Ex. 1001, 1:4–10. In this process, monoethylenically unsaturated monomers, such as acrylic acid, methacrylic acid, and acrylamide, are mixed with an initiator of polymerization, preferably in the form of a water soluble azo initiator, and polymerized to form a viscoelastic, gelatinous polymer. *Id.* at 3:24–46, 5:29–34, 9:37–39 (disclosing the production of a “solid, viscoelastic and only slightly sticky” polymer).

The ’329 patent contains one Figure, reproduced below, that depicts the disclosed reactor schematically:



As shown in the Figure, the reactor of the ’329 patent is generally composed of “tubular reactor (1) which has a conical taper (2) at the end” and “shut-off element (6).” *Id.* at 2:26–28, 2:63–64. D1 represents the diameter of tubular reactor 1, D2 represents the diameter of conical taper 2 at the end of the taper, and angle α represents the angle between D1 at the start of the conical taper and the inner cone wall. *Id.* at 2:52–60. The ratio between D1 and D2 is between 2:1 and 25:1 and the angle α “is >45° and <90°, and preferably from 65° to 85°.” *Id.* at 2:17–23, 2:52–60.

In the disclosed process, a monomer solution is mixed with an initiator of polymerization at point 3 and fed into tubular reactor 1. *Id.* at 2:28–32. After the monomers are polymerized, an inert gas is provided via feeds 4 and 8 to “completely” push the polymerized material out of the reactor. *Id.* at 2:46–48, 3:3–6 (“Instead of a single feed line (8), it can be advantageous if a multiplicity of openings are arranged in the form of a circle at the end of the cone”), 7:1–3 (“the gel can then be completely pushed out of the reactor after opening the shut-off element.”).

C. Illustrative Claims

Claims 1 and 4 of the ’329 patent are illustrative of the challenged claims and are reproduced below:

> 1. A process for preparing high molecular weight polymers, which comprises polymerizing water-soluble, monoethylenically unsaturated monomers and, if desired, crosslinkers which contain at least two nonconjugated, ethylenically unsaturated double bonds in the molecule, and, if desired, water-insoluble monoethylenically unsaturated monomers in aqueous solution in the presence of polymerization initiators in a tubular reactor which has a conical taper at the end, the ratio of the diameter of the reactor (D1) to the diameter at the end of the conical taper of the reactor (D2) being from 2:1 to 25:1 and the angle between D1 at the start of the conical taper and the inner cone wall being >45° and <90°, and *removing the gelatinous reaction mixture by injection of an inert gas*.

Ex. 1001, 14:27–41 (emphasis added).

> 4. A process as claimed in claim 1, wherein the water soluble monoethylenically unsaturated monomers are polymerized with from 0.001 to 5% by weight, based on the total monomers employed in the polymerization, of at least one crosslinker.

Id. at 14:49–53.

D. Instituted Grounds of Unpatentability

We instituted the instant trial based on the following grounds of unpatentability (Inst. Dec. 17):

1. Whether claims 1–3 and 5–7 would have been obvious under 35 U.S.C. § 103 over the ’944 patent[1] and the ’597 patent; [2] and

2. Whether claim 4 would have been obvious under 35 U.S.C. § 103 over the ’944 patent, the ’597 patent, and EP ’709.[3]

In support of its grounds of unpatentability, Petitioner also relies upon the testimony of Dr. Benny Freeman. Ex. 1007.

II. ANALYSIS

A. Claim Construction

Because the ’329 patent has expired, the parties agree that the claims are to be given their ordinary and customary meaning in accordance with the claim construction principles set forth in *Phillips v. AWH Corp.*, 415 F.3d 1303 (Fed. Cir. 2015) (en banc). PO Resp. 8 (citing *In re Rambus, Inc.*, 753 F.3d 1253, 1256 (Fed. Cir. 2014)); Tr. 9:18–21.

In the Institution Decision we construed the term “by injection of an inert gas” to require that the inert gas act directly upon the gelatinous reaction mixture. Inst. Dec. 7. The parties do not dispute this construction in their subsequent briefing and, upon review of the record as a whole, we discern no reason to modify this construction. *See* PO Resp. 8–9; Pet. Reply 3–6.

In the Patent Owner Response, Patent Owner requests that we also construe the terms “removing” and “the gelatinous reaction mixture.” PO Resp. 9–14. Upon review of the parties’ arguments and supporting

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[1] U.S. Patent No. 3,634,944, iss. Jan. 18, 1972 (Ex. 1004).
[2] U.S. Patent No. 3,784,597, iss. Jan. 8, 1974 (Ex. 1002).
[3] EP Patent Publication No. 0374709 A2, published June 27, 1990 (Ex. 1006).

evidence, we determine that only the term "removing" requires construction for the purposes of this Decision. *See Vivid Techs., Inc. v. Am. Sci. & Eng'g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999) (only terms in controversy need to be construed, and only to the extent necessary to resolve the controversy).

removing

Claim 1 requires "removing the gelatinous reaction mixture by injection of an inert gas." Ex. 1001, 14:40–41. Petitioner asserts the ordinary meaning of the term "removing" in this claim phrase is "discharging." Pet. 19. Petitioner's sole support for this construction, however, is the testimony of Dr. Freeman, who provides no supporting evidence or analysis to explain why one of ordinary skill in the art would understand "removing" to mean "discharging." *See* Ex. 1007 ¶¶ 103–104.

In its response, Patent Owner contends that "removing" should be construed to require that "substantially all the gelatinous reaction mixture is discharged from the reactor." PO Resp. 9, 12 (citing Ex. 2016 ¶¶ 79–83). Patent Owner asserts this construction is supported by the ordinary meaning of the term "removing," which is "to get rid of: eliminate," and the Specification of the '329 patent, which indicates that one of the problems the inventor was attempting to solve was the "incomplete discharge of polymer gel when using gas pressure." *Id.* at 9–11 (citing Ex 2001; Ex. 2002; Ex. 1001, 1:36–41); *CVI/Beta Ventures, Inc. v. Tura LP*, 112 F.3d 1146, 1160 (Fed. Cir. 1997) ("In construing claims, the problem the inventor was attempting to solve, as discerned from the specification and the prosecution history, is a relevant consideration.")). Patent Owner also asserts that this construction is consistent with the examples set forth in the '329 patent, which indicate that the polymer gel was discharged from the tubular reactor either "without residue" or with "only insignificant" residue remaining in the reactor. PO Resp. 12 (citing Ex. 1001, 8:63–14:25).

In Reply, Petitioner maintains its position that "removing" means "discharging," but contends that Patent Owner's declarant, Dr. John Carson, admitted during cross-examination that the term "removing" is broad enough to encompass 90% discharge of a polymer gel. Pet. Reply 3 (citing Ex. 1017, 120:10–14). Petitioner notes that it "does not object to" to this construction, because there is allegedly "no dispute that the prior art '944 and '597 patents removed at least 90% [of the polymerized material]." *Id.*

Upon review of the record as a whole, we are persuaded by Patent Owner's argument that the term "removing"—in the context of the claim phrase "removing the gelatinous reaction mixture by injection of an inert gas"—means "substantially all the gelatinous reaction mixture is discharged from the reactor." First, as noted by Patent Owner, the terms "remove" and "discharge" have different meanings—with "discharge" defined as "to pour forth fluid or other contents" and "remove" defined as "to get rid of: eliminate." *See* Ex. 2001 (definition of "discharge"); Ex. 2002 (definition of "remove").

Second, the terms "discharge" and "remove" are used differently in the '329 patent. For example, the '329 patent distinguishes one prior art reference on the basis that, although the reference discloses the "discharge" of polymer from a reactor using inert gas pressure, "it was not possible in this way completely to remove the polymer from the reactor." Ex. 1001, 1:33–41; Ex. 1014 ¶¶ 2–3.

Finally, the '329 patent uses the term "remove" in other contexts to indicate the elimination of substances from the reactor. Specifically, the

’329 patent notes that because the polymerization reaction is to be carried out “in the absence of oxygen,” an inert gas is passed through the monomer and initiator solutions “*to remove* residual oxygen.” Ex. 1001, 6:32–38 (emphasis added).

Although “removing” indicates elimination of the polymer from the reactor, the parties agree that the term does not preclude an “insignificant amount” of residual polymer from remaining in the reactor. Ex. 2003, 162:19–163:7 (Dr. Freeman testifying that “ideally you would remove all the material that you made. But, I don’t think the ’329 patent requires absolutely [sic] removal of all of the material. And the examples, certainly there is one of them at least that shows that there is some residue.”); Ex. 1001, 14:23–24 (noting that in Example 14 “[o]nly insignificant residues remained in the tubular reactor”). The ’329 patent does not indicate, however, what level of residual polymer constitutes an “insignificant” amount.

The only extrinsic evidence identified by the parties specifically identifying a range of residual polymer allowed by the “removing” claim term is the testimony of Dr. Carson, who testifies as follows:

> Q. What percentage of removal is required, in your opinion?
>
> A. I believe that Dr. Schork, in his declaration, stated virtually all the material, which doesn’t mean 100 percent, but essentially all of the material, with a small amount of residue that could be left on the interior surface.
>
> Q. Does that require, say, 90 percent?
>
> A. I –
>
> MR. RIDDLE: Objection to form.
>
> A. I would say that it’s considerably more than 90 percent, probably higher than 95, 98, 99 percent.

> BY MR. BRADY:
>
> Q. So removal, according to the ’329 patent, then, in your opinion, would require removal of 90 percent, at least, of the gelatinous reaction mixture. Is that your testimony?
>
> A. Yes.

Ex. 1017, 119:18–120:14.

Petitioner contends this testimony demonstrates that discharging 90% of a polymer from a reactor would satisfy the “removing” limitation of claim 1. Pet. Reply 3. Contrary to Petitioner’s argument, however, Dr. Carson’s testimony indicates that discharge levels “considerably more than 90%, probably higher than 95, 98, and 99 percent” (which would necessarily require that at least 90% of the material be discharged) are required to satisfy the “removing” limitation. Thus, given that Dr. Carson’s testimony is the only evidence bearing on the point, we are not persuaded that Petitioner directs us to sufficient evidence to demonstrate that a 90% discharge level would satisfy the “removing” limitation in claim 1 of the ’329 patent.

B. Principles of Law

A patent claim is unpatentable under 35 U.S.C. § 103(a) if the differences between the claimed subject matter and the prior art are such that the subject matter, as a whole, would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. *KSR Int’l Co. v. Teleflex Inc.*, 550 U.S. 398, 406 (2007). The question of obviousness is resolved on the basis of underlying factual determinations, including: (1) the scope and content of the prior art; (2) any differences between the claimed subject matter and the prior art;

(3) the level of ordinary skill in the art;[4] and (4) if in the record, objective evidence of nonobviousness. *Graham v. John Deere Co.*, 383 U.S. 1, 17–18 (1966).

C. Obviousness over the ’944 Patent and the ’597 Patent

Petitioner contends that claims 1–3 and 5–7 would have been obvious over the ’944 patent and the ’597 patent. Pet. 44–51.

1. The ’944 Patent

The ’944 patent is directed to the production and drying of polyacrylamide polymer gels, which the ’944 patent reports are “intensely adhesive” and “so viscous as to be self-supporting.” Ex. 1004, 1:4–9, 1:15–17, 1:22–27. Figure 1A of the ’944 patent is reproduced below:

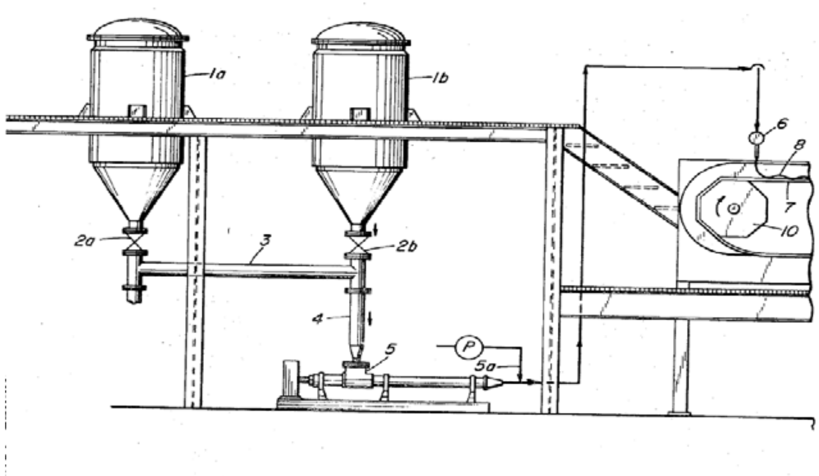

FIG. 1A

Figure 1A “is an elevation showing schematically the reactors” of the ’944 patent.

In this figure, tubular polymerization chambers 1a and 1b have a conical taper and supply an acrylamide polymer gel to screw pump 5 through valves 2a and 2b and pipes 3 and 4. *Id.* at 3:23–28. The gel is then

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[4] The level of ordinary skill in the art in this case is reflected by the prior art of record. *See Okajima v. Bourdeau*, 261 F.3d 1350, 1355 (Fed. Cir. 2001).

supplied under the pressure provided by screw pump 5 to header 6 and extruded on steel belt 7 in the form of cords 8. *Id.* at 3:28–31, 4:68–74.

2. The ’597 Patent

Similar to the ’944 patent, the ’597 patent also discloses a process for producing a “viscous” and “sticky” acrylamide polymer gel. Ex. 1002, 1:21–24, 1:50–51. Although the ’597 patent does not contain depictions of the reactor used to polymerize the acrylamide monomers, in Example 1 of the ’597 patent the reaction vessel is described as eight centimeters in diameter, fifty centimeters in height, and having an outlet two centimeters in diameter at the bottom of the vessel. *Id.* at 3:60–62. In Example 2, the reaction vessel is described as being sixty centimeters in diameter, two-hundred centimeters in height, and having an outlet ten centimeters in diameter at the bottom of the vessel. *Id.* at 4:34–36.

The ’597 patent discloses that the acrylamide polymer may be “discharged” “by use of nitrogen gas pressure.” *Id.* at 3:70–72. The ’597 patent also discloses that “[a]ny conventional reaction vessel may be used” in the process, but notes that reaction vessels having a “bottom product outlet for discharging the gel-like polymer containing medium” are preferred. *Id.* at 2:70–3:2.

3. The Parties’ Arguments

Petitioner contends that each limitation of the challenged claims is taught or suggested by the combined teachings of the ’944 and ’597 patents. Pet. 44–51. For example, Petitioner contends that the ’944 patent discloses discharging “sticky” gels from a tubular reaction vessel having a conical taper that is less than 90 degrees and greater than 45 degrees. *Id.* at 44–45 (citing Ex. 1007 ¶¶ 227, 238). Petitioner further contends that, because the

’597 patent explicitly teaches successfully discharging a sticky polymer gel from a reaction vessel using inert gas pressure, it would have been obvious to likewise use inert gas pressure in the ’944 patent to discharge the polymer gel from the disclosed reactor. *Id.* at 20–22, 45–46 (citing Ex. 1002, 2:70–3:2, 3:70–72). According to Petitioner, “[c]ombining the disclosed features of the ’944 and ’597 patents would have been merely the combination of known elements according to known methods to yield predic[t]able results.” *Id.* at 47 (citing Ex. 1007 ¶ 236).

Patent Owner responds that Petitioner has not shown that the recited prior art references teach every element of the challenged claims. PO Resp. 2–3. In particular, Patent Owner contends that the ’944 and ’597 patents do not disclose that inert gas pressure can be used to *remove* a sticky, self-supporting polymer gel from a reactor. *Id.* at 3, 23–24.

In Reply, Petitioner asserts that “[t]here is no dispute that the prior art ’944 and ’597 patents removed at least 90%” of the polymer gel from the reactor. Pet. Reply 3. Petitioner further argues that U.S. Patent No. 5,081,215 (“the ’215 patent”), which is admitted prior art disclosed in the ’329 patent, teaches that a conical taper and inert gas pressure can be used to discharge a polymer gel from a reaction vessel “virtually without residue.” *Id.* at 2–3, 11.

4. Analysis

To demonstrate unpatentability, Petitioner must either present evidence that each limitation of the claimed invention is disclosed in the recited prior art, or identify “some reason why one of skill in the art would modify the prior art to obtain the claimed invention.” *Nike, Inc. v. Adidas AG*, 812 F.3d 1326, 1335 (Fed. Cir. 2016); *PAR Pharm., Inc. v. TWi Pharm.,* *Inc.*, 773 F.3d 1186, 1194 (Fed. Cir. 2014) ("We must first determine whether TWi carried its burden to prove that all claimed limitations are disclosed in the prior art.") (quoting *Medichem, S.A. v. Rolabo, S.L.*, 437 F.3d 1157, 1164 (Fed. Cir. 2006)).

As noted above, Patent Owner contends that Petitioner has not carried its burden to show that the '597 and '944 patents, either individually or in combination, disclose "*removing* a gelatinous reaction mixture by injection of an inert gas." We agree.

The '944 patent discloses discharging a "sticky" polymer gel from a tubular reactor having a conical taper at one end. Pet. 44 (citing Ex. 1004, Fig. 1A). The '597 patent discloses "discharging" this same type of polymer gel from a tubular reactor using inert gas pressure. *Id.* at 45; Ex. 1002, 2:71–3:2, 3:70–73. Neither patent discloses, however, the extent of polymer discharge. And, although Dr. Freeman testifies that one of ordinary skill in the art would understand that the proposed combination of the '944 and '597 patents would result in removal of the polymer gel from the reactor (Ex. 1007 ¶¶ 231–235), this testimony presumes that "discharging" is equivalent to "removing." *Id.* ¶¶ 103–104 (asserting that "a process of discharging resulting gelatinous material from the reactor will satisfy [the removing] element of the claim"), 120, 232. Thus, we are not persuaded that the Petition demonstrates, by a preponderance of the evidence, that all elements of the challenged claims are disclosed in the asserted prior art.

In Reply, Petitioner asserts that even under Patent Owner's proposed construction the '944 and '597 patents disclose "removing" a gelatinous reaction mixture using inert gas pressure because "[t]here is no dispute that the prior art '944 and '597 patents removed at least 90% [of the polymer]."

Pet. Reply. 3. We are not persuaded by this argument for at least two reasons. First, the 90% discharge threshold relied upon by Petitioner is premised on a construction of the term "removing" that we do not adopt. Second, Petitioner cites to no evidentiary support for its assertion that the prior art '944 and '597 patents achieve 90% discharge, nor does Petitioner cite to persuasive evidence to establish that the combined teachings of the '944 and '597 patents would necessarily[5] result in discharge of "substantially all" of the polymer gel from the reactor. *See id*; *see also PAR Pharm.*, 773 F.3d 1194–95 (noting that "inherency may supply a missing claim limitation in an obviousness analysis" if the limitation is "necessarily present" or is the "natural result" of the combination of prior art elements).

Petitioner also argues that "[a] high expectation of success" in achieving the claimed invention "is provided by the explicit '597 disclosure of inert gas and D1:D2 ratios, the broad D1:D2 range of the '329 claims, which permits the discharge opening to be up to one-half the diameter of the reactor itself, and the prior art directly rebutting Dr. Schork." Pet. Reply 14. Again, this argument is premised on a construction of "removing" that requires only 90% discharge. *See id.* ("As Dr. Carson confirmed, the '329 claims only require removing at least 90% of the gel mixture."). Moreover, Petitioner does not explain adequately why the D1:D2 ratios of the '597 patent would necessarily result in removal of the polymer gel from the

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[5] Petitioner does not assert in the Petition or Reply that the combined teachings of the '944 and '597 patents would inherently result in "removing" the polymer gel from the respective reaction vessels. *See* Tr. 14:3–13. Thus, Petitioner has not met the "high standard" for demonstrating inherency in the context of an obviousness analysis. *See Par Pharm.*, 773 F.3d at 1195–96.

reactor, or why a relatively large diameter discharge opening would ensure that only an insignificant amount of polymer remains in the reaction vessel. As such, on this record we do not find Petitioner's unsupported arguments persuasive.

During oral argument, Petitioner also identified paragraph 125 of Dr. Freeman's reply declaration as demonstrating that "one of ordinary skill in the art would expect that the process [of the '597 patent] discharged most, if not all, of the polymer formed in the reactor." Tr. 17:23–8:5 (answering a question raised at Tr. 15:7–16). In this paragraph, Dr. Freeman testifies:

> . . . I see no evidence in the '597 patent that less than all of the material in the reactor was discharged in either Example 1 or Example 2. Furthermore, as the '597 patent describes the downstream process, one skilled in the art would expect that the '597 patent describes a process that discharges most, if not all, of the polymer formed in the reactor.

Ex. 1019 ¶ 125.

We decline to consider this testimony because Petitioner did not cite to it or explain its significance in the Reply. *See* 37 C.F.R. §§ 42.6(a)(3) (noting that "[a]rguments must not be incorporated by reference from one document into another"), 42.23(b) ("All arguments for the relief requested in a motion must be made in the motion."). Nevertheless, even if we were to consider this testimony, it would be entitled to little, if any, weight because Dr. Freeman does not explain *why* one of ordinary skill in the art, observing that the '597 patent focuses primarily on the downstream processing of the polymer gel and does not recite a level of discharge, would expect that "most, if not all, of the polymer" is discharged from the reactor. *See Rohm and Haas Co. v. Brotech Corp.*, 127 F.3d 1089, 1092 (Fed. Cir. 1997) (noting that a fact finder is not required to "credit the unsupported assertions of an expert witness"); *Ashland Oil, Inc. v. Delta Resins & Refractories, Inc.*, 776 F.2d 281, 294 (Fed. Cir. 1985) (noting that the "[l]ack of factual support" for an expert opinion "may render the testimony of little probative value").

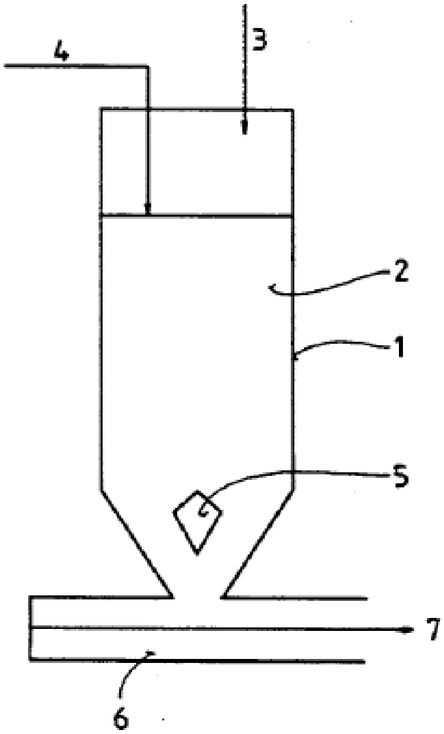
Petitioner also argues in the Reply that the '329 patent admits that U.S. Patent No. 5,081,215 discloses using inert gas pressure to discharge a polymer gel "virtually without residue." Pet. Reply 11 (citing Ex. 1001, 2:1–4; 1020, 4:2–3, 5:31–35); Tr. 19:11–14. This disclosure is as follows:

> U.S. Pat. No. 5,081,215 discloses a process for preparing polyether ketones in which the polycondensation is carried out by a two-stage process, the polycondensation being completed in the second stage and the plastic polymer matrix containing included aluminum chloride particles obtainable being discharged from the tubular reactor virtually without residue by injecting an inert gas.

Ex. 1001, 1:65–2:4.

A patentee's representations as to "what is to be considered prior art in determining obviousness of their improvement" may be taken as a binding admission. *In re Nomiya*, 509 F.2d 566, 570–71 (CCPA 1975); *Constant v. Advanced Micro-Devices Inc.*, 848 F.2d 1560, 1570 (Fed. Cir. 1988) ("A statement in a patent that something is in the prior art is binding on the applicant and patentee for determinations of anticipation and obviousness."). In this case, however, we are not persuaded that Petitioner establishes the purported admission.

The '215 patent discloses a "process for the preparation of polyaryleneetherketones by electrophilic polycondensation in two reaction zones." Ex. 1020, 1:5–7. The '215 patent contains one Figure, which is reproduced below:



The Figure is a schematic view of the tube reactor of the ’215 patent.

As shown in the Figure, the downstream reactor of the ’215 patent is composed of reaction tube 1, reaction material 2, displacement apparatus 5, and discharge aid 6. Ex. 1020, 3:55–63. In the process of the ’215 patent, inert gas pressure, preferably in the form of nitrogen, is used to force the reaction material “towards the discharge aid in the conical part of the reactor. From there, the reaction material is discharged *by the discharge aid* into a downstream working-up unit.” *Id.* at 4:45–50 (emphasis added); *see also id.* at 4:37–38 (noting that inert gas pressure is used to move “the reaction material with plug flow to the discharge aid in the lower part of the reactor”), 5:46–50 (noting that the discharge screw is “mounted in the lower, conical part of the downstream reactor”).

Given that the ’215 patent discloses the use of both inert gas pressure and a discharge aid located within the conical portion of the reactor to discharge the sticky polymer from the reaction vessel, we do not understand the statement in the ’329 patent that inert gas pressure was used to discharge the polymer material “virtually without residue” to be a binding admission that it was known in the prior art that gas pressure and a conical taper alone will discharge a polymer from a reaction vessel without residue.[6] Indeed, the use of a discharge aid in the ’215 patent reactor tends to support Patent Owner’s argument that one of ordinary skill in the art would not have had a reasonable expectation of success in using a conical taper and inert gas pressure alone (the combination proposed by Petitioner) to remove a sticky, gelatinous product from a reactor.

5. Conclusion

Based on the foregoing, and upon review of the record as a whole, we determine that there is insufficient evidence or argument to demonstrate that the ’944 and ’597 patents disclose, or would inherently result in, “removing the gelatinous reaction mixture by injection of an inert gas.” Thus, we are not persuaded that Petitioner has demonstrated, by a preponderance of the evidence, that claims 1–3 and 5–7 would have been obvious over the ’944 and ’597 patents.

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[6] Although the ’329 patent claims do not appear to preclude the use of a screw pump within the reactor, Petitioner does not make this argument, and this is not the combination of prior art elements proposed in the Petition. *See Intelligent Bio-Systems, Inc. v. Illumina Cambridge Ltd.*, 821 F.3d 1359, 1369 (Fed. Cir. 2016) (noting that “the expedited nature of IPRs bring with it an obligation for petitioners to make their case in their petition to institute”).

D. Obviousness of Claim 4 over the ’944 Patent, the ’597 Patent, and EP ’709

Petitioner contends that claim 4 of the ’329 patent would have been obvious over the combined teachings of the ’944 patent, the ’597 patent, and EP ’709. Pet. 57–59; Inst. Dec. 16. Because claim 4 depends from claim 1, and because Petitioner does not contend that EP ’709 bears on the issue of whether “substantially all” of the polymer gel would be removed from the reaction vessel, we are also not persuaded that Petitioner has demonstrated, by a preponderance of the evidence, that claim 4 would have been obvious over the ’944 patent, the ’597 patent, and EP ’709.

E. Motion to Exclude

Patent Owner moves to exclude Exhibits 1003, 1005, and 1020–1031, as well as those portions of Dr. Freeman’s reply declaration (Ex. 1019) that rely upon the identified exhibits. Paper 37, 1–12. As we have either not relied upon these exhibits, or have considered the exhibits and found in favor of Patent Owner (e.g., Ex. 1020), we dismiss the Motion to Exclude as moot.

F. Motion to Exclude Errata

On March 30, 2016, Patent Owner requested authorization to replace unsigned deposition transcripts (Exhibits 1018 and 2003) with signed versions of these transcripts, including errata sheets. Petitioner opposed this request because the errata sheets would change the deponent’s substantive testimony rather than address only transcription errors. Upon review of the proposed errata sheets, we agreed with Petitioner that the errata sheet for Exhibit 1018 (the transcript of Joseph Schork) would modify substantive testimony of the declarant; however, in order to maintain a complete record in this proceeding we permitted Patent Owner to file the signed declaration testimony with accompanying errata sheets and authorized Petitioner to file a motion to exclude the new exhibits. Paper 41, 3. Petitioner now seeks to exclude this testimony. Paper 43.

As noted by Petitioner, errata sheets that seek to change the substantive testimony of a witness are disfavored. *See Rules of Practice for Trials Before the Patent Trial and Appeal Board*, 77 Fed. Reg. 48612, 48642 (Aug. 14, 2012) (noting in Comment 144 that the Board's final rules do not provide for errata sheets, although a party may seek to submit an errata if necessary to the proceeding); *Garmin Int'l, Inc. v. Cuozzo Speed Techs LLC*, IPR2012-0001, Paper 50 at 3 (PTAB July 18, 2013) ("a request to make a material change to the substance of cross examination testimony is unlikely to be successful no matter when the request is made.").

In this case, we need not determine whether this is one of the rare instances where errata testimony that changes the substance of a declarant's testimony may be relied upon by a party, as we have not relied upon any of the disputed testimony for purposes of this decision. Accordingly, we dismiss Petitioner's motion to exclude the errata sheets as moot.

G. Patent Owner's Identification of Allegedly Improper Reply Evidence

Pursuant to authorization from the Board, Patent Owner filed a notice identifying arguments and evidence that it believes "exceed the proper scope of reply under 37 C.F.R. § 42.23(b)." Paper 31, 1. We have considered Patent Owner's identifications, as well as Petitioner's opposition (Paper 32), and find that we have either not relied upon any of the allegedly improper reply evidence, or have considered the evidence and found in favor of Patent

Owner. Accordingly, we do not strike or ignore any of the identified arguments and evidence.

H. Patent Owner's Demonstrative Exhibits

Prior to oral argument in this proceeding, Petitioner filed an objection to Patent Owner's demonstrative exhibits. Paper 47 (identifying slides 5, 13, 19, 26, 27, 29, 30–32, 40–42, 51, 69, and 70). In its filing, Petitioner contends Patent Owner's demonstrative exhibits contain numerous slides with citations to testimony and evidence that were not otherwise cited in any paper. *Id.* at 2–4. During oral argument, and in support of these slides, Patent Owner asserted:

> Deposition testimony is testimony like it would be in District Court under the Federal Rules. It's considered testimony at the proceeding . . . My understanding is there wouldn't be a problem in doing that. Needless to say, it's not a new argument or new evidence . . . [as] all of these issues we brought up in our response and in declaration and during depositions.

Tr. 59:5–19.

In our Order setting oral argument, we directed the parties to *St. Jude Medical, Cardiology Division, Inc. v. The Board of Regents of the University of Michigan*, IPR2013-00041 (PTAB January 27, 2014) (Paper 65) for guidance regarding the appropriate content of demonstrative exhibits. Paper 40, 3. As noted in *St. Jude*, demonstrative exhibits "are intended to be visual aids to assist the party in making its oral presentation." Paper 65, 3. Demonstrative exhibits are not evidence, however, and "cannot add new evidence to the record of the proceeding," nor can demonstrative exhibits "rely on evidence that, although it is in the record, was never specifically discussed in any paper before the Board." *Id.*

Here, Patent Owner concedes that several of the slides identified by Petitioner contain citations to declaration and deposition testimony that were not previously discussed or cited to in any paper submitted by the parties. *See* Tr. 59:8–23. As such, these slides are contrary to the guidance provided in our Order. Paper 40, 3. Accordingly, we have excluded from our consideration Patent Owner's demonstrative slides 5, 13, 19, 26, 27, 29, 30–32, 40–42, 51, 69, and 70.

III. ORDER

For the foregoing reasons, it is:

ORDERED that no claim of the '329 patent is held unpatentable;

FURTHER ORDERED that Patent Owner's Motion to Exclude is *dismissed as moot*;

FURTHER ORDERED that Petitioner's Motion to Exclude Errata is *dismissed as moot*; and

FURTHER ORDERED that, because this is a Final Written Decision, parties to the proceeding seeking judicial review of the decision must comply with the notice and service requirements of 37 C.F.R. § 90.2.

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US005633329A

United States Patent [19]

Hähnle et al.

[11] **Patent Number: 5,633,329**

[45] **Date of Patent: May 27, 1997**

[54] **PREPARATION OF HIGH MOLECULAR WEIGHT POLYMERS**

[75] Inventors: **Hans-Joachim Hähnle**, Ludwigshafen; **Eckhard Neufeld**, Limburgerhof; **Gunnar Schornick**, Neuleiningen; **Roland Minges**, Grünstadt; **Thomas Anstock**, Weisenheim; **Jürgen Tropsch**, Römerberg; **Hans-Jürgen Krauss**, Mannheim, all of Germany

[73] Assignee: **BASF Aktiengesellschaft**, Ludwigshafen, Germany

[21] Appl. No.: **591,315**

[22] Filed: **Jan. 25, 1996**

[30] **Foreign Application Priority Data**

Jan. 31, 1995 [DE] Germany 195 02 939.9

[51] **Int. Cl.**^{6} **C08F 2/18**

[52] **U.S. Cl.** **526/64**; 526/317.1; 526/329.7

[58] **Field of Search** 526/64, 317.1, 526/329.7

[56] **References Cited**

U.S. PATENT DOCUMENTS

5,081,215	1/1992	Koch et al.	528/125

FOREIGN PATENT DOCUMENTS

101893	3/1984	European Pat. Off. .
0101893	3/1984	European Pat. Off. .
0374709	6/1990	European Pat. Off. .
1218157	6/1966	Germany .
119354	4/1976	Germany .
05057181	8/1993	Japan .

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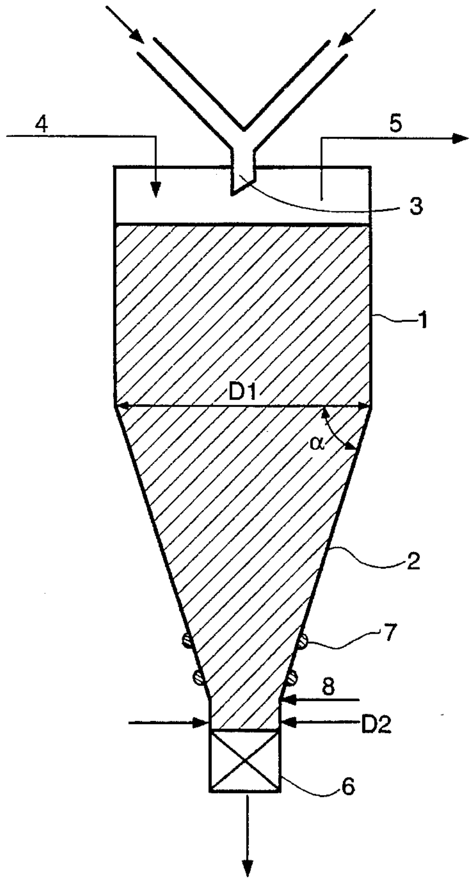
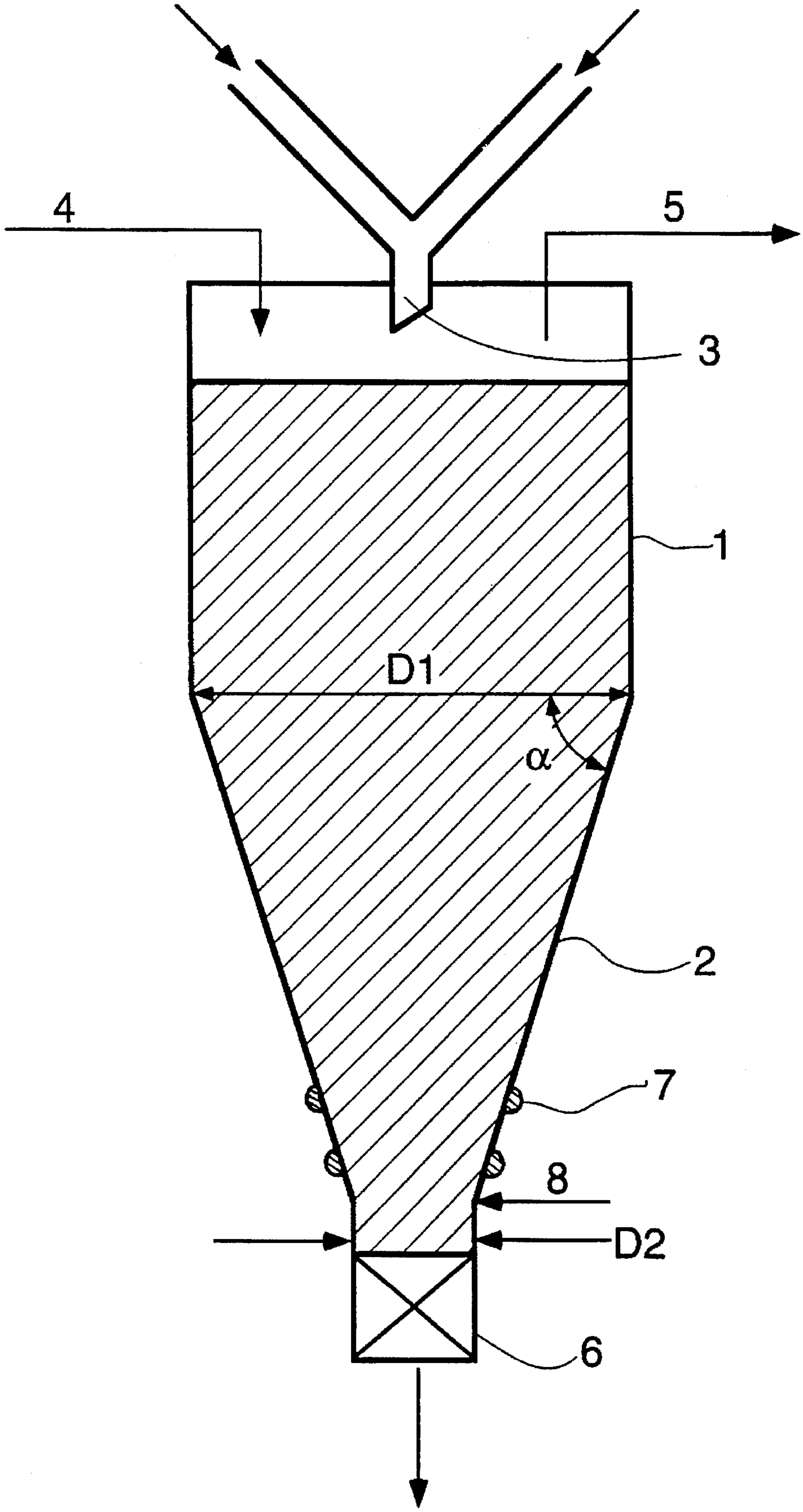
Attorney, Agent, or Firm—Oblon, Spivak, McClelland, Maier & Neustadt, P.C.

[57] **ABSTRACT**

Process for the preparing high molecular weight polymers by polymerization of water-soluble, monoethylenically unsaturated moonomers and, if desired, crosslinkers, and, if desired, water-insoluble monoethylenically unsaturated monomers, in aqueous solution in the presence of polymerization initiators in a tubular reaction which at the end has a conical taper, the ratio of the diameter of the reactor (D1) to the diameter at the end of the conical taper of the reactor (D2) being from 2:1 to 25:1 and the angle between D1 at the beginning of the conical taper and the inner cone wall being >45° and <90°, and discharge of the gelatinous reaction mixture from the reactor by injection of an inert gas.

7 Claims, 1 Drawing Sheet

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PREPARATION OF HIGH MOLECULAR WEIGHT POLYMERS

The invention relates to a process for preparing high molecular weight polymers by polymerization of water-soluble, monoethylenically unsaturated monomers and, if desired, crosslinkers which contain at least two nonconjugated, ethylenically unsaturated double bonds in the molecule, and, if desired, water-insoluble monoethylenically unsaturated monomers in aqueous solution in the presence of polymerization initiators in a tubular reactor and removal of the gelatinous reaction mixture from the reactor by injection of an inert gas.

DE-B-12 18 157 discloses a process for preparing water-soluble polymers by polymerization of water-soluble, monoethylenically unsaturated monomers in aqueous solution in the presence of polymerization initiators in a cylindrical reactor which is provided with closable connections. Polymerization takes place batchwise. The resulting gelatinous reaction mixture is discharged from the cylindrical reactor with the aid of a fitted piston which can be moved along the cylinder axis.

JP-A-93/57181 likewise discloses a tubular reactor having a movable piston running against the wall for ejecting the polymer gels obtainable during polymerization. The disadvantage of such tubular reactors equipped with a movable piston can be seen in the high wear which is caused during operation by friction between the inner wall of the reactor and the piston. As the polymer gels are frequently of sticky consistency, as a rule very smooth or else wall surfaces with an adhesion-decreasing coating are needed, which are particularly susceptible to the wear described. Moreover, as indicated in said Japanese application as prior art, it was known to discharge polymer gels from a reactor lined with Teflon by injection of an inert gas such as nitrogen. In this process, however, the inert gas prematurely escapes from the reactor through a gap which is formed between the inner wall of the reactor and the gel before the polymer has been pressed through the outlet opening completely. It is therefore not possible in this way completely to remove the polymer from the reactor.

EP-B 0 374 709 discloses a continuous process for preparing liquid-absorbent, crosslinked, water-insoluble polymers by polymerization of water-soluble monomers in a tubular reactor in the presence of polymerization initiators in aqueous medium to give polymer gels. In this process, a separating liquid which is immiscible with this solution is fed in together with the aqueous monomer solution at the reactor inlet, the separating liquid being automatically distributed between the reactor inner wall and polymer phase during the course of the polymerization and the resulting monomers together with the separating liquid being discharged at the reactor outlet. The polymer can be removed from the reactor, for example, by an increased pressure of an inert gas. The breakthroughs of the monomer mixture occurring through the gelatinous polymer or along the interface between reactor wall and polymer gel are also problematic here again in this process. In the case of a breakthrough of the monomer mixture through the polymer gel, the polymerization must be interrupted and the equipment cleaned. The use of separating agents does reduce the number of breakthroughs through the gel, but is associated with the disadvantage that the separating agent remains in the reaction product.

U.S. Pat. No. 5,081,215 discloses a process for preparing polyether ketones in which the polycondensation is carried out by a two-stage process, the polycondensation being

completed in the second stage and the plastic polymer matrix containing included aluminum chloride particles obtainable being discharged from the tubular reactor virtually without residue by injecting an inert gas.

It is an object of the present invention to make available an improved process for preparing high molecular weight polymers which form a polymer gel in aqueous medium.

We have found that this object is achieved by a process for preparing high molecular weight polymers by polymerization of water-soluble, monoethylenically unsaturated monomers and, if desired, crosslinkers which contain at least two nonconjugated, ethylenically unsaturated double bonds in the molecule, and, if desired, water-insoluble monoethylenically unsaturated monomers in aqueous solution in the presence of polymerization initiators in a tubular reactor and removal of the gelatinous reaction mixture from the reactor by injection of an inert gas, if the tubular reactor has a conical taper at the end, the ratio of the diameter of the reactor (D1) to the diameter at the end of the conical taper of the reactor (D2) being from 2:1 to 25:1 and the angle between D1 at the start of the conical taper and the inner cone wall being >45° and <90°. The angle between D1 and the inner cone wall is preferably from 65° to 85°.

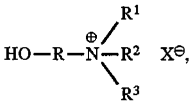
BRIEF DESCRIPTION OF THE DRAWING

Polymerization is carried out in a reactor which is shown diagrammatically in the figure. This is essentially a tubular reactor (1) which has a conical taper (2) at the end. Before entry of the reaction mixture to be polymerized, the aqueous monomer solution and the initiator solution, which are customarily prepared and fed to the reactor separately from one another, are mixed (3). Suitable equipment for this purpose is any which is suitable for rapid mixing of low viscosity liquids, eg. static mixers.

The tubular reactor (1) consists essentially of a vertical tube of circular cross-section. The inner cylindrical surface of the tube is preferably lined with a material which is inert and anti-adhesive to the reaction mixture. Polytetrafluoroethylene, for example, is suitable for this purpose. The tube body (1) preferably has a ratio of height:diameter of from 4 to 40 and in particular from 6 to 15. The equipment can be sealed pressure-tight. At the top of the tube body are provided one or more feeds for the reaction mixture and for an inert gas (4). In some cases it can be advantageous to use a mixture of inert gas and solvent for pressing out the gelatinous polymer from the reactor. The solvents can be introduced, for example, through the inert gas line (4) or separately therefrom at the top of the reactor. At the top of the reactor a feed line (5) is additionally present, via which the pressure in the tubular reactor can be reduced. The lower end of the tubular reactor has a conical taper (2). The ratio of the diameter of the reactor (D1) to the diameter at the end of the conical taper of the reactor (D2) is from 2:1 to 25:1 and is preferably in the range from 2:1 to 20:1. A very particularly preferred ratio of D1:D2 is from 3:1 to 15:1. The conical taper of the reactor (2) is such that the angle α between D1 at the start of the conical taper and the inner cone wall is >45° and <90°, preferably from 65° to 85°. For most practical applications the angle α is from 75° to 85°.

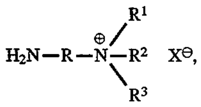
The reactor can be surrounded by a jacket such that the reaction mixture situated therein can be heated or cooled from outside. At the end of the conical taper is located a shut-off element (6). To the shut-off element can be attached an outlet tube, which at its free end, if desired, has a further taper whose dimensions are optimized to the distribution of the gel to be discharged. At the end of the cone is located an arrangement for radioactive level measurement (**7**), and at least one connection (**8**) through which inert gas can additionally be introduced into the reactor. Instead of a single feed line (**8**), it can be advantageous if a multiplicity of openings are arranged in the form of a circle at the end of the cone. The polymer gels discharged from the reactor are then worked up in a customary manner, ie. they can be further divided, eg. with the aid of a cutting knife, and the comminuted gel then dried. Equipment suitable for this purpose is the customary equipment such as tumble dryers, paddle dryers, belt dryers, a stirred solid bed or a fluidized bed.

High molecular weight polymers are obtained by the process according to the invention by polymerization of water-soluble, monoethylenically unsaturated monomers and, if desired, crosslinkers which contain at least two nonconjugated, ethylenically unsaturated double bonds in the molecule, and, if desired, water-insoluble monoethylenically unsaturated monomers. If the polymerization is carried out in the absence of crosslinkers, water-soluble polymers are formed. The amount of water-insoluble monoethylenically unsaturated monomers, which, if desired, are additionally used in the polymerization, is at most so high that copolymers which are still water-soluble result. The water-soluble monoethylenically unsaturated monomers are designated in the following as monomers of group (a). Suitable monomers of this group are, for example, ethylenically unsaturated C_3- to C_6-carboxylic acids, their amides and esters with aminoalcohols of the formula


$$HO-R-\overset{\oplus}{N}(R^1)(R^2)(R^3)\quad X^{\ominus},$$

(I)

where $R=C_2$- to C_5- alkylene, R^1, R^2, R^3=H, CH_3, C_2H_5, C_3H_7 and $X^{\ominus}$ is an anion. Additionally suitable are amides which are derived from amines of the formula


$$H_2N-R-\overset{\oplus}{N}(R^1)(R^2)(R^3)\quad X^{\ominus},$$

(II)

The substituents in formula II and $X^{\ominus}$ have the same meanings as in formula I.

These compounds are, for example, acrylic acid, methacrylic acid, crotonic acid, itaconic acid, maleic acid, fumaric acid, acrylamide, methacrylamide, crotonamide, dimethylaminoethyl acrylate, diethylaminoethyl acrylate, dimethylaminoneopentyl acrylate and dimethylaminoethyl methacrylate, dimethylaminopropyl acrylate, dimethylaminoneopentyl acrylate and dimethylaminoneopentyl methacrylate. The basic acrylates and methacrylates or basic amides which are derived from the compounds of the formula II are employed in the form of the salts with strong mineral acids, sulfonic acids or carboxylic acids or in quaternized form. The anion $X^{\ominus}$ for the compounds of the formula I is the acid radical of the mineral acids or of the carboxylic acids or methosulfate, ethosulfate or halide from a quaternizing agent.

Further water-soluble monomers of group (a) are N-vinylpyrrolidone, N-vinylformamide, acrylamidopropanesulfonic acid, vinylphosphonic acid and/or alkali metal or ammonium salts of vinylsulfonic acid. The other acids can likewise be employed in the polymerization either in unneutralized form or in partially or up to 100% neutralized form. Suitable water-soluble monomers of group (a) are also diallylammonium compounds, such as dimethyldiallylammonium chloride, diethyldiallylammonium chloride or diallylpiperidinium bromide, N-vinylimidazolium compounds, such as salts or quaternization products of N-vinylimidazole and 1-vinyl-2-methylimidazole, and N-vinylimidazolines, such as N-vinylimidazoline, 1-vinyl-2-methylimidazoline, 1-vinyl-2-ethylimidazoline or 1-vinyl-2-n-propylimidazoline, which are likewise employed in the polymerization in quaternized form or as a salt.

Preferred monomers of group (a) are acrylic acid, methacrylic acid, sodium or potassium acrylate, sodium or potassium methacrylate, acrylamide or dimethylaminoethyl acrylate in quaternized form or as a salt or mixtures of the monomers. These monomers can be copolymerized with one another in any desired ratio, acrylic acid partially neutralized using sodium hydroxide solution, monomer mixtures of acrylamide and acrylic acid and/or sodium acrylate and monomer mixtures of acrylamide and dimethylaminoethyl acrylate methochloride being particularly preferably employed.

In order to prepare crosslinked polymers which are used as super-absorbers, at least one monomer of group (a) is polymerized with at least one monomer of group (b). In the following, crosslinkers designated as a monomer of group (b) are those which contain at least two nonconjugated, ethylenically unsaturated double bonds. Suitable crosslinkers are, for example, N,N'-methylenebisacrylamide, polyethylene glycol diacrylates and polyethylene glycol dimethacrylates, which are in each case derived from polyethylene glycols of a molecular weight from 126 to 8500, preferably 400 to 2000, trimethylolpropane triacrylate, trimethylolpropane trimethacrylate, ethylene glycol diacrylate, propylene glycol diacrylate, butanediol diacrylate, hexanediol diacrylate, hexanediol dimethacrylate, diacrylates and dimethacrylates of block copolymers of ethylene oxide and propylene oxide, polyhydric alcohols di- or triesterified with acrylic acid or methacrylic acid, such as glycerol or pentaerythritol, triallylamine, tetraallylethylenediamine, divinylbenzene, diallyl phthalate, polyethylene glycol divinyl ethers of polyethylene glycols of a molecular weight from 126 to 4000, trimethylolpropane diallyl ether, butanediol divinyl ether, pentaerythritol triallyl ether and/or divinylethyleneurea. Preferably, water-soluble crosslinkers are employed, eg. N,N'-methylenebisacrylamide, polyethylene glycol diacrylates, polyethylene glycol dimethacrylates, pentaerythritol triallyl ether and/or divinylurea. The monomers of group (b) are only used in the preparation of water-insoluble polymers, namely in amounts from 0.001 to 5, preferably from 0.01 to 2.0, % by weight, based on the total monomers employed in the copolymerization.

The copolymerization of the monomers of groups (a) and (b) can additionally be carried out, if an alteration of the properties of the crosslinked copolymers is desired, in the presence of water-insoluble, monoethylenically unsaturated monomers, in the following designated by monomers of group (c). Monomers of group (c) are, for example, hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxyethyl methacrylate, hydroxypropyl methacrylate, acrylonitrile and/or methacrylonitrile. Additionally suitable are esters of acrylic acid or methacrylic acid with monohydric alcohols containing 1 to 18 carbon atoms, eg. methyl acrylate, ethyl acrylate, propyl acrylate, isopropyl acrylate, n-butyl acrylate, isobutyl acrylate, hexyl acrylate, 2-ethylhexyl acrylate, stearyl acrylate, the corresponding esters of methacrylic acid, diethyl fumarate, diethyl maleate, dimethyl maleate, dibutyl maleate, vinyl formate, vinyl acetate and vinyl propionate. If the monomers of group (c) are used for the modification of the water-soluble or the water-insoluble polymers, they are employed in amounts from 0.5 to 20, preferably from 2 to 10, % by weight, based on the total monomers taking part in the polymerization.

If desired, the polymerization can be carried out in the presence of the customary polymerization regulators. Suitable polymerization regulators are, for example, thio compounds, such as thioglycolic acid, mercaptoalcohols, eg. 2-mercaptoethanol, mercaptopropanol and mercaptobutanol, dodecylmercaptan, formic acid, ammonia and amines, eg. ethanolamine, diethanolamine, triethanolamine, triethylamine, morpholine and piperidine.

The monomers (a) and, if desired, (b) and, if desired, (c) are polymerized in from 10 to 80, preferably from 20 to 60, % strength by weight aqueous solution in the presence of polymerization initiators. The polymerization initiators employed can be all compounds which dissociate into radicals under the polymerization conditions, eg. peroxides, hydroperoxides, hydrogen peroxide, persulfates, azo compounds and redox catalysts. The use of water-soluble initiators is preferred. In some cases it is advantageous to use mixtures of various polymerization initiators, eg. mixtures of hydrogen peroxide and sodium or potassium peroxodisulfate. Mixtures of hydrogen peroxide and sodium peroxodisulfate can be used in any desired ratio. Suitable organic peroxides are, for example, acetylacetone peroxide, methyl ethyl ketone peroxide, tert-butyl hydroperoxide, cumene hydroperoxide, tert-amyl perpivalate, tert-butyl perpivalate, tert-butyl perneohexanoate, tert-butyl perisobutyrate, tert-butyl per-2-ethylhexanoate, tert-butyl perisononanoate, tert-butyl permaleate, tert-butyl perbenzoate, tert-butyl per-3,5,5-trimethylhexanoate and tert-amyl perneodecanoate. Particularly suitable polymerization initiators are water-soluble azo initiators, eg. 2,2'-azobis-(2-amidinopropane) dihydrochloride, 2,2'-azobis(N,N'-dimethylene) isobutyramidine dihydrochloride, 2-(carbamoylazo) isobutyronitrile, 2,2'-azobis[2-(2'-imidazolin-2-yl)propane] dihydrochloride and 4,4'-azobis(4-cyanovaleric acid). Said polymerization initiators are employed in customary amounts, eg. in amounts from 0.01 to 5, preferably 0.1 to 2.0, % by weight, based on the monomers to be polymerized.

As oxidizing component, the redox catalysts contain at least one of the abovementioned percompounds and, as reducing component, for example ascorbic acid, glucose, sorbose, ammonium or alkali metal hydrogensulfite, sulfite, thiosulfate, hyposulfite, pyrosulfite or sulfide, metal salts, such as iron(II) ions or silver ions or sodium hydroxymethylsulfoxylate. The reducing component of the redox catalyst preferably used is ascorbic acid or sodium sulfite. Based on the amount of monomers employed in the polymerization, from 3×10^{-6} to 1 mol % of the reducing component of the redox catalyst system and from 0.001 to 5.0 mol % of the oxidizing component of the redox catalyst, for example, are used. Instead of the oxidizing component of the redox catalyst, one or more water-soluble azo starters can also be used.

In the preparation of water-soluble polymers which are used, for example, as flocculating agents, the polymerization of the monomers of group (a) described above is carried out in the absence of monomers of group (b) indicated above. If desired, the monomers of group (c) can additionally be used for the modification of the water-soluble polymers.

For the preparation of water-absorbent polymers, ie. polymers which are insoluble in water but swell therein, the water-soluble monoethylenically unsaturated monomers of group (a) are polymerized with from 0.001 to 5.0% by weight, based on the total monomers employed in the polymerization, of at least one crosslinker. The crosslinkers are described above as monomers of group (b). The water-insoluble polymers can also be modified, if desired, with the monomers of group (c). Crosslinked polyacrylic acids and crosslinked polyacrylamides are especially of industrial interest as superabsorbers.

For the preparation of flocculating agents, acrylic acid partially neutralized with sodium hydroxide solution, monomer mixtures of acrylamide and acrylic acid and/or sodium acrylate and monomer mixtures of acrylamide and dimethylaminoethyl acrylate methochloride are particularly preferably polymerized. The monomers can be copolymerized with one another in any desired ratio. It is also possible to polymerize them to homopolymers, eg. homopolymers of acrylamide and homopolymers of dimethylaminoethyl acrylate methochloride.

The polymerization is carried out in each case in aqueous solution or in solvent mixtures which contain at least 50% by weight of water. Suitable water-miscible solvents are, for example, glycols such as ethylene glycol, propylene glycol and butylene glycol, and polyethylene glycols of a molecular weight of up to 4000, and methyl and ethyl ethers of glycols and polyglycols.

The polymerization of the monomers of group (a) on their own and, if appropriate, in the presence of the monomers (c) results in water-soluble polymers. The preparation of water-insoluble polymers by polymerizing the monomers of group (a) and the monomers of group (b) and, if desired, the monomers of group (c) in aqueous solution in the presence of polymerization initiators results in polymer gels. The monomers and the initiators are dissolved, for example, in stirring vessels in the aqueous medium to be polymerized. If desired, the initiators can be fed to the reactor in the form of a solution in an organic solvent. The solution of the monomers and the initiators is preferably adjusted to a temperature in the range from –20° to 30+ C. In order to remove residual oxygen from the solution of the monomers and the initiators, an inert gas is customarily passed through these solutions. Inert gases suitable for this purpose are, for example, nitrogen, carbon dioxide or rare gases such as neon or helium. The polymerization is carried out in the absence of oxygen. The solutions of the monomers and of the initiator are mixed with one another before they reach the reactor, the introduction of monomers and initiator into the tubular reactor preferably being carried out in a countercurrent of an inert gas. The polymerization can be carried out batchwise or continuously. In a batchwise procedure, the reactor shown in the figure, for example, is filled with an aqueous monomer solution and a solution of the initiator. As soon as the polymerization starts, the reaction mixture warms up depending on the selected starting conditions, such as concentration of the monomers in the aqueous solution and nature of the monomers. On account of the heat of polymerization released, the temperature of the reaction mixture rises to, for example, from 30 to 180, preferably 40° to 130° C. The polymerization can be carried out at normal pressure, under reduced pressure or even at elevated pressure. Working at elevated pressure can be advantageous in those cases when the temperature maximum to be expected in the polymerization is above the boiling point of the solvent mixture used. On the other hand, it can be advantageous especially in the preparation of very high molecular weight products to lower the maximum temperature with the aid of vapor cooling by polymerizing under reduced pressure. The tubular reactor is in most cases jacketed such that the reaction mixture can be cooled or heated as required. After completion of the polymerization reaction, the polymer gel obtained can be rapidly cooled, for example, by cooling the reactor wall.

In order to discharge the polymer gel from the tubular reactor; an inert gas is injected onto the polymer gel at the top of the tubular reactor and the gel can then be completely pushed out of the reactor at the end of the reactor after opening the shut-off element. If desired, the polymer gel can also be discharged from the reactor by injecting an inert liquid, which is preferably a precipitating agent for the polymer, at the top of the tubular reactor. In order to discharge the resulting polymer gel from the reactor, a pressure from 2 to 65, preferably from 4 to 25, bar, for example, is necessary. The pressure data relate both to an inert gas and to an inert liquid which are introduced at the top of the reactor to discharge the polymer gel. As a result of the pressure increase at the top of the reactor, the gelatinous product is discharged from the reactor in a plug flow. Plug flow in this context is understood as meaning a flow in which a uniform falling of the surface of the gel plug takes place, virtually no deformation of the surface of the gel plug occurring. In order to discharge the polymer gel from the reactor, the pressure with which the gel is transported can be reduced in one or more stages. In the figure, a pressure reduction in one stage is shown diagrammatically. The pressure reduction is carried out by suitable selection of the ratio of the diameters **D1** and **D2**. If desired, the transport pressure can be reduced in a further or in several subsequent pressure stages. Depending on the composition of the polymer gel and on the pressure used for ejecting the gel, the gel can break down into individual particles without mechanical comminution on letting down to normal pressure. If desired, the gel coming from the reactor can be further comminuted, as indicated above, in a subsequent processing unit, dried and, if necessary, freed from residual monomers.

The polymerization is carried out adiabatically in batchwise operation. High molecular weight products are obtained by the process according to the invention. The molecular weights of the water-soluble products are above 100,000 and are preferably from 1×10^6 to 20×10^6. They have K values according to Fikentscher of from 180 to 300 (determined in 5% strength aqueous sodium chloride solution at a polymer concentration of 0.1% by weight and a temperature of 25° C). No K value can be given for the cross-linked polymers, because the crosslinked polymers do not dissolve in water or another solvent. A molecular weight determination is not possible for the crosslinked polymers.

The water-soluble polymers are used, for example, as flocculating agents in industrial and municipal sewage treatment plants, as retention agents in the production of paper and as thickeners for aqueous systems. The crosslinked polymers have a high absorption power for water and are accordingly used as superabsorbers, eg. in diapers.

The percentage data in the examples are percentages by weight if not stated otherwise. The K values of the polymers were determined according to H. Fikentscher, Zellulose-Chemie (Cellulose Chemistry), Volume 13 (1932), 58–64 and 71–74. The measurements were carried out in 5% strength by weight sodium chloride solution at a polymer concentration of 0.1% by weight, a temperature of 25° C. and a pH of 7.

The determination of the charge density was carried out according to D. Horn., Polyethylenimine—Physicochemical Properties and Application, (IUPAC) Polymeric Amines and Ammonium Salts, Pergamon Press Oxford and New York, 1980, pages 333–355.

The absorption capacity of the superabsorber for water per gram of superabsorber was determined with the aid of the teabag test. The liquid used here is a 0.9% strength sodium chloride solution. A defined amount of gelatinous, water-absorbent copolymer (1 g) is filled into a teabag, which is then closed. The dimensions of the teabag must be appropriately matched to the amount of gelatinous copolymer weighed in. The teabag is then immersed in the liquid for a specified time and then reweighed after a drainage period of 15 seconds. To calculate the absorption capacity, a blank test must be carried out in which a teabag without gelatinous, water-absorbent copolymer is immersed in the solution and the weight of the teabag is determined after the drainage period indicated above. The absorption capacity then results from the following relationship

$$\text{Absorption capacity} = \frac{\text{Weight of the teabag with polymer gel} - \text{weight of the teabag in the blank test}}{\text{Weight of the polymer gel weighed in}}$$

The retention is determined as follows: The same procedure as above, only instead of drainage of the teabags centrifugation is carried out for 3 min at 1400 rpm in a centrifuge having a diameter of 230 mm.

$$\text{Retention} = \frac{\text{Weight of the teabag after centrifuging} - \text{weight of the teabag in the blank test}}{\text{Weight of the polymer gel weighed in}}$$

The loss due to washing out is determined by dispersing the water-insoluble polymer in a 0.9% strength by weight sodium chloride solution, stirring the dispersion for 16 hours, then filtering and titrimetrically determining the amount of the extracted fraction in the filtrate.

EXAMPLES

Example 1

Preparation of a Superabsorber

In a vessel designated by **1**, an aqueous solution was prepared from 1362.5 g of distilled water, 8363.5 g (32.92 mol) of a 37% strength aqueous sodium acrylate solution, 789.85 g (11.0 mol) of acrylic acid, 19.43 g of trimethylolpropane triacrylate and 64.75 g of a 15% strength aqueous sodium peroxodisulfate solution. This solution was temperature controlled at 20° C. Nitrogen was passed through the solution for 20 minutes and a solution of 0.097 g of ascorbic acid in 499.9 g of distilled water was prepared simultaneously in a second vessel, likewise temperature controlled at 20° C. and nitrogen was passed through the solution for 20 minutes. After preparation of the solutions, the contents of the two vessels were injected into the reactor synchronously under a pressure of 2 bar in a nitrogen countercurrent, both solutions being mixed with the aid of a static mixer before entry into the reactor at the site designated by **3** in the figure.

The reactor had a length of 1000 mm, a tube diameter D1 of 160 mm and tapered conically at the end to a diameter D2 of 50 mm. The ratio D1/D2 was 3.2. The cone length was 320 mm and the angle α indicated in the figure in the isosceles triangle between D1 at the beginning of the conical taper and the inner cone wall was 80°.

After the mixing of the two aqueous solutions described above, the polymerization started immediately. The nitrogen flushing of the reactor was stopped and the tubular reactor was closed. The reaction mixture reached a maximum temperature of 99° C. It was allowed to cool to room temperature overnight. A nitrogen pressure of 9 bar was injected at the top of the reactor via the line designated by **4** in the figure. After opening the shut-off valve **6**, it was possible to discharge all the gelatinous reactor contents without residue, the product dividing into irregularly shaped gel particles of a few centimeters diameter at the outlet from an outlet tube having a diameter of 100 mm, not shown in the figure. The solid viscoelastic, barely sticky gel thus obtained was further comminuted using a cutting knife and then dried at 70° C. in a drying oven at 20 mbar for 15 hours. The absorption capacity for water was 45 g/g of polymer; the retention 33 g of water per g of polymer. The loss due to washing out was 22%.

Example 2

Preparation of a Superabsorber

Example 1 was repeated with the only exception that the polymerization was carried out under a pressure of 8 bar. The maximum temperature of the reaction mixture reached in this case was 95° C. The consistency of the gel obtained was solid, viscoelastic and barely sticky. It was possible to discharge the polymer gel from the tubular reactor without residue under a pressure of 9 bar. The absorption capacity was 46 g of water per g of polymer, the retention 34 g of water per g of polymer and the loss due to washing out 26%.

Example 3

Preparation of a Superabsorber

Starting from the following mixture:

Vessel **1**:
Distilled water: 807.7 g
Sodium acrylate solution 37% in water: 8843.4 g (35.8 mol)
Acrylic acid: 857.5 g (11.9 mol)
Trimethylolpropane triacrylate: 21.1 g
Sodium peroxodisulfate solution 15% in water: 70.3 g

Vessel **2**:
Distilled water: 499.9 g
Ascorbic acid: 0.105 g

The mixture was polymerized as described in Example 2. The maximum temperature reached was 112° C. The consistency of the gel obtained was solid, viscoelastic and only slightly sticky. It was possible to discharge the product without residue at a pressure of 9 bar. The polymer had the following properties:

Absorption capacity: 44 g/g
Retention: 32 g/g
Loss due to washing out: 21%.

Example 4

Preparation of a Superabsorber

Starting from the following mixture:

Vessel **1**:
Distilled water: 118.5 g
Sodium acrylate solution 37% in water: 9482.1 g (37.6 mol)
Acrylic acid: 903.2 g (12.5 mol)
Trimethylolpropane triacrylate: 22.2 g
Sodium peroxodisulfate solution 15% in water: 74.0 g

Vessel **2**:
Distilled water: 499.9 g
Ascorbic acid: 0.111 g

The mixture was polymerized as described in Example 2. The maximum temperature reached was 127° C. The consistency of the gel obtained was solid, viscoelastic and only slightly sticky. It was possible to discharge the product without residue at a pressure of 9 bar. The polymer had the following properties:

Absorption capacity: 40 g/g
Retention: 27 g/g
Loss due to washing out: 17%.

Example 5

Preparation of a Superabsorber

Starting from the following mixture:

Vessel **1**:
Distilled water: 1362.5 g
Sodium acrylate solution 37% in water: 8363.5 g (32.9 mol)
Acrylic acid: 789.9 g (11.0 mol)
Trimethylolpropane triacrylate: 19.4 g

Vessel **2**:
Distilled water: 497.66 g
2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 2.34 g

The mixture was polymerized as described in Example 1. The maximum temperature reached was 97° C. The consistency of the gel obtained was solid, viscoelastic and only slightly sticky. It was possible to eject the product from the tubular reactor without residue at an overpressure of 9 bar. The polymer had the following properties:

Absorption capacity: 44 g/g
Retention: 34 g/g
Loss due to washing out: 18%.

Example 6

Preparation of an Anionic Flocculating Agent

Solutions were prepared from the following constituents in the two vessels **1** and **2**.

Vessel **1**:
Distilled water: 5454.7 g
Acrylamide solution 50% in water: 2304.0 g (16.2 mol)
Sodium acrylate solution 37.3% in water: 1737.3 g (6.9 mol)
10% strength aqueous solution of diethylenetriaminepentasodium acetate: 4.0 g

Vessel **2**:
Distilled water: 498.9 g
2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 1.08 g

Both solutions were temperature controlled at 25° C. Nitrogen was passed through the solutions for 30 minutes. The contents of the two vessels were then injected synchronously into the tubular reactor described in Example 1 at an elevated pressure of 2 bar in a nitrogen countercurrent. After commencement of the exothermic reaction, the nitrogen flushing was stopped and the tubular reactor was closed. The polymerization started within a short time after mixing. The reaction mixture reached a maximum temperature of 68° C. It cooled to room temperature overnight. A solid, visoelastic, barely sticky gel was obtained. A conically tapered tube 150 mm long lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to eject the gel through the nozzle having an opening of 10 mm without problems at pressures from 4 to 8 bar. No residues remained in the tubular reactor after ejection. The gel extrudates obtained were comminuted and dried in a vacuum drying oven at 50° C. The polymer had a K value of 287 and a charge density of 3.2 meq/g.

Example 7

Preparation of an Anionic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and sodium acrylate was prepared as described in Example 6.

Vessel **1**:
Distilled water: 4273.9 g
Acrylamide solution 50% in water: 400.0 g (2.8 mol)
Sodium acrylate solution 37.3% in water: 4825.7 g (19.1 mol)
10% strength aqueous solution of diethylenetriaminepentasodium acetate: 0.4 g

Vessel **2**:
Distilled water: 498.8 g
2,2'-Azobis[2(2'-imidazolin2-yl)propane]dihydrochloride: 1.2 g

The consistency of the gel was soft, viscoelastic and not very sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to eject the gel through the nozzle having an opening of 10 mm without residue at pressures between 4 and 6 bar at the inlet of the reactor. The polymer had a K value of 244 and a charge density of 9.6 meq/g.

Example 8

Preparation of an Anionic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and sodium acrylate was prepared as described in Example 6. However, after injection of the solutions into the tubular reactor a pressure of 60 mbar was set and maintained during the polymerization and the cooling phase.

Vessel **1**:
Distilled water: 1244.2 g
Acrylamide solution 50% in water: 364.0 g (2.6 mol)
Sodium acrylate solution 37.3% in water: 4391.4 g (12.4 mol)
10% strength aqueous solution of diethylenetriaminepentasodium acetate: 0.36 g

Vessel **2**:
Distilled water: 498.9 g
2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 1.09 g

The consistency of the gel was more solid than that of the polymer described in Example 6, viscoelastic and not very sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to discharge the gel through the nozzle having an opening of 10 mm without residue at a pressure of 6 bar. The polymer had a K value of 244 and a charge density of 9.6 meq/g.

Example 9

Preparation of an Anionic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and sodium acrylate was prepared as described in Example 8.

Vessel **1**:
Distilled water: 2526.1 g
Acrylamide solution 50% in water: 2600.0 g (18.3 mol)
Sodium acrylate solution 37.3% in water: 871.3 g (3.5 mol)
10% strength aqueous solution of diethylenetriaminepentasodium acetate: 2.6 g

Vessel **2**:
Distilled water: 499.0 g
2.2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 0.98 g

The consistency of the gel obtained was solid, viscoelastic and not very sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to discharge the gel through the nozzle having an opening of 20 mm without residue at a pressure from 6 to 7 bar. The copolymer had a K value of 284 and a charge density of 2.7 meq/g.

Example 10

Preparation of a Nonionic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and sodium acrylate was prepared as described in Example 6.

Vessel **1**:
Distilled water: 5835.3 g
50% strength aqueous acrylamide solution: 3420.0 g (24.1 mol)
37.3% strength aqueous sodium acrylate solution: 241.3 g (0.9 mol)
10% strength aqueous solution of diethylenetriaminepentasodium acetate: 3.42 g

Vessel **2**:
Distilled water: 498.9 g
2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 1.08 g

The consistency of the gel thus obtained was solid, viscoelastic and not very sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to discharge the gel through the nozzle having an opening of 10 mm without residue by injection of 5 to 7 bar of nitrogen at the inlet of the reactor through the line designated by **4** in the figure. The copolymer had a K value of 261.

Example 11

Preparation of a Cationic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and 2-trimethylammoniumethylacryL late chloride was prepared as described in Example 6.

Vessel **1**:
Distilled water: 7050.8 g
50% strength aqueous acrylamide solution: 2797.2 g (19.7 mol)
80% strength aqueous solution of 2-trimethylammoniumethyl acrylate chloride: 749.3 g (3.1 mol)
10% strength aqueous solution of diethylenetriaminepentasodium acetate: 2.80 g

Vessel **2**:
Distilled water: 498.75 g
2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 1.25 g

The consistency of the gel was still solid, viscoelastic and slightly sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to eject the gel through the 10 mm nozzle without residue at a pressure from 5 to 7 bar. The copolymer had a K value of 262 and a charge density of 2.1 meq/g.

Example 12

Preparation of a Cationic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and 2-trimethylammoniumethylacrylate chloride was prepared as described in Example 6.

Vessel **1**:

Distilled water: 6418.3 g

50% strength aqueous acrylamide solution: 2664.0 g (18.7 mol)

80% strength aqueous solution of 2-trimethylammoniumethyl acrylate chloride: 1665.0 g (6.9 mol)

10% strength aqueous solution of diethylenetriaminepentasodium acetate: 2.66 g

Vessel **2**:

Distilled water: 348.6 g

2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 1.67 g

The consistency of the gel was soft, viscoelastic and slightly sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to discharge the gel through the nozzle having an opening of 5 mm without residue at a pressure from 4 to 7 bar. The copolymer had a K value of 255 and a charge density of 2.9 meq/g.

Example 13

Preparation of a Cationic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and 2-trimethylammoniumethylacrylate chloride was prepared as described in Example 6. However, the mixture was ejected at a temperature of 50° C. 2 hours after reaching the maximum temperature.

Vessel **1**:

Distilled water: 7501.3 g

50% strength aqueous acrylamide solution: 1998.0 g (14.1 mol)

80% strength aqueous 2-trimethylammoniumethyl acrylate chloride solution: 1248.8 g (5.2 mol)

10% strength aqueous solution of diethylenetriaminepentasodium acetate: 2.0 g

Vessel **2**:

Distilled water: 348.75 g

2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 1.25 g

The consistency of the gel was soft, viscoelastic and slightly sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 5 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to discharge the gel through the nozzle having an opening of 5 mm without residue at a pressure from 3 to 6 bar. The copolymer had a K value of 253 and a charge density of 3.0 meq/g.

Example 14

Preparation of a Cationic Flocculating Agent

Starting from the following mixture, a further high molecular weight copolymer of acrylamide and 2-trimethylammoniumethylacrylate chloride was prepared as described in Example 6.

Vessel **1**:

Distilled water: 5999.0 g

50% strength aqueous acrylamide solution: 1000.0 g (7.0 mol)

80% strength aqueous 2-trimethylammoniumethyl acrylate chloride solution: 2500.0 g (10.3 mol)

10% strength aqueous solution of diethylenetriaminepentasodium acetate: 1.0 g

Vessel **2**:

Distilled water: 347.5 g

2,2'-Azobis[2-(2'-imidazolin-2-yl)propane]dihydrochloride: 2.5 g

The consistency of the gel was soft, viscoelastic and sticky. A conically tapered tube lined with Teflon, which at the end had an inner diameter of 10 mm, was attached to the shut-off element at the outlet of the reactor described in Example 1. It was possible to discharge the gel through the nozzle having an opening of 10 mm at a nitrogen pressure from 3 to 5 bar at the inlet of the reactor. Only insignificant residues remained in the tubular reactor. The copolymer had a K value of 239 and a charge density of 3.9 meq/g.

We claim:

1. A process for preparing high molecular weight polymers, which comprises polymerizing water-soluble, monoethylenically unsaturated monomers and, if desired, crosslinkers which contain at least two nonconjugated, ethylenically unsaturated double bonds in the molecule, and, if desired, water-insoluble monoethylenically unsaturated monomers in aqueous solution in the presence of polymerization initiators in a tubular reactor which has a conical taper at the end, the ratio of the diameter of the reactor (D1) to the diameter at the end of the conical taper of the reactor (D2) being from 2:1 to 25:1 and the angle between D1 at the start of the conical taper and the inner cone wall being >45° and <90°, and removing the gelatinous reaction mixture by injection of an inert gas.

2. A process as claimed in claim **1**, wherein the angle between D1 and the inner cone wall is from 65° to 85°.

3. A process as claimed in claim **1**, wherein the water-soluble monoethylenically unsaturated monomers employed are acrylic acid, methacrylic acid, sodium or potassium acrylate, sodium or potassium methacrylate, acrylamide or dimethylaminoethyl acrylate in quaternized form or as a salt or mixtures of the monomers.

4. A process as claimed in claim **1**, wherein the water-soluble monoethylenically unsaturated monomers are polymerized with from 0.001 to 5% by weight, based on the total monomers employed in the polymerization, of at least one crosslinker.

5. A process as claimed in claim **1**, wherein the polymerization initiators employed are water-soluble azo initiators.

6. A process as claimed in claim **1**, wherein the polymer gel is removed at the end of the reactor by injection of an inert gas at the entrance of the reactor at a pressure of from 2 to 65 bar.

7. A process as claimed in claim **1**, wherein the polymer gel is removed at the end of the reactor by injection of an inert gas at the entrance of the reactor at a pressure of from 4 to 25 bar.

* * * * *

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Paper 9
Entered: August 6, 2015

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

SNF HOLDING COMPANY, FLOPAM INC.,
CHEMTALL INCORPORATED, SNF SAS, and
SNF (CHINA) FLOCCULANT CO. LTD.,
Petitioner,

v.

BASF CORPORATION,
Patent Owner.

Case IPR2015-00600
Patent 5,633,329

Before, GRACE KARAFFA OBERMANN, JON B. TORNQUIST, and JEFFREY W. ABRAHAM, *Administrative Patent Judges.*

TORNQUIST, *Administrative Patent Judge*.

DECISION
Institution of *Inter Partes* Review
37 C.F.R. § 42.108

I. INTRODUCTION

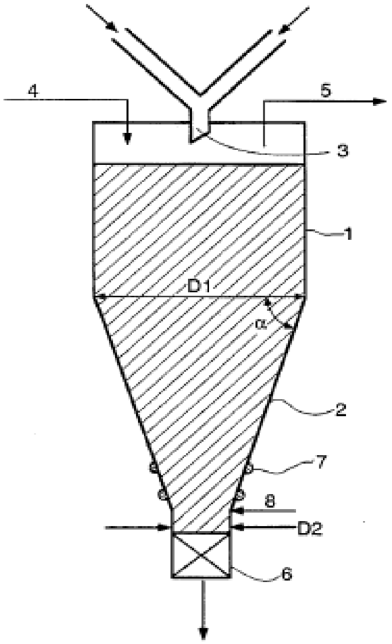
SNF Holding Company, Flopam Inc., Chemtall Incorporated, SNF SAS, and SNF (China) Flocculant Co. Ltd. (collectively "Petitioner") filed a Petition (Paper 1) requesting institution of *inter partes* review of claims 1–7 of U.S. Patent No. 5,633,329 (Ex. 1001, "the '329 patent"). BASF Corporation ("Patent Owner") filed a Preliminary Response (Paper 8, "Prelim. Resp.") to the Petition.

We have jurisdiction under 35 U.S.C. § 314(a), which provides that an *inter partes* review may not be instituted "unless . . . there is a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition." For the reasons given below, we determine that Petitioner has demonstrated a reasonable likelihood of prevailing with respect to claims 1–7 of the '329 patent. Accordingly, we authorize an *inter partes* review to be instituted as to these claims on the grounds set forth below.

A. The '329 Patent

The '329 patent relates to a process for polymerizing water-soluble, monoethylenically unsaturated monomers in an aqueous solution. Ex. 1001, 1:4–10. Suitable monoethylenically unsaturated monomers may include, for example, acrylic acid, methacrylic acid, and acrylamide. *Id.* at 3:24–46. In the disclosed process, an initiator of polymerization is used, preferably in the form of a water soluble azo initiator. *Id.* at 5:29–34. The disclosed process results in a "sticky" gelatinous polymer mixture, which is removed from the reactor "by injection of an inert gas." *Id.* at 1:11–13, 1:29–32, 6:66–7:6.

The ’329 patent contains one Figure, which is depicted below:



The Figure of the ’329 patent depicts the polymerization reactor, including tubular reactor 1, conical taper 2, and inert gas feed 4. *Id.* at 2:26–44. D1 represents the diameter of tubular reactor 1; D2 represents the diameter of conical taper 2 at the end of the taper, and angle α represents the angle between D1 at the start of the conical taper and the inner cone wall. *Id.* at 2:18–20, 2:52–60.

In the disclosed process, the ratio between D1 and D2 is between 2:1 and 25:1 and the angle α “is >45° and <90°, and preferably from 65° to 85°.” *Id.* at 2:17–23, 2:52–60.

1. Illustrative Claim

Claim 1, set forth below, is the only independent claim of the ’329 patent and is illustrative of the challenged claims:

> 1. A process for preparing high molecular weight polymers, which comprises polymerizing water-soluble, monoethylenically unsaturated monomers and, if desired, crosslinkers which contain at least two nonconjugated, ethylenically unsaturated double bonds in the molecule, and, if desired, water-insoluble monoethylenically unsaturated monomers in aqueous solution in the presence of polymerization initiators in a tubular reactor which has a conical taper at the end, the ratio of the diameter of the reactor (D1) to the diameter at the end of the conical taper of the reactor (D2) being from 2:1 to 25:1 and the angle between D1 at the start of the conical taper and the inner cone wall being >45° and <90°, and removing the gelatinous reaction mixture by injection of an inert gas.

Ex. 1001, 14:27–41.

2. *Related Proceeding*

The parties agree that Patent Owner has asserted the ’329 patent against Petitioner in the Southern District of Texas in an action captioned *BASF Corporation v. SNF Holding Company*, Civ. No. 4:14-cv-02733 (S.D. Tex.). Pet. 1; Paper 6, 2.

B. *The Asserted Grounds*

Petitioner asserts the following grounds of unpatentability:

1. Whether claims 1–3 and 5–7 are unpatentable under 35 U.S.C. § 103 as having been obvious over the ’597 patent[1] and GB ’028;[2]

2. Whether claims 1–3 and 5–7 are unpatentable under 35 U.S.C. § 103 as having been obvious over the ’597 patent, GB ’028, and the ’460 patent;[3]

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[1] U.S. Patent No. 3,784,597 (Jan. 8, 1974) (Ex. 1002)
[2] GB Patent No. 1,054,028 (Published Jan 4, 1967) (Ex. 1005)
[3] U.S. Patent No. 2,918,460 (Dec. 22, 1959) (Ex. 1003)

3. Whether claims 1–3 and 5–7 are unpatentable under 35 U.S.C. § 103 as having been obvious over the ’944 patent[4] and the ’597 patent;

4. Whether claims 1–3 and 5–7 are unpatentable under 35 U.S.C. § 103 as having been obvious over the ’944 patent, the ’597 patent, and GB ’028;

5. Whether claims 1–3 and 5–7 are unpatentable under 35 U.S.C. § 103 as having been obvious over the’944 patent, the ’597 patent, GB ’028, and the ’460 patent;

6. Whether claims 4–5 are unpatentable under 35 U.S.C. § 103 as having been obvious over the ’597 patent, GB ’028, the ’460 patent, and EP ’709;[5] and

7. Whether claims 4–5 are unpatentable under 35 U.S.C. § 103 as having been obvious over the ’944 patent, GB ’028, the ’460 patent, and EP ’709.

Pet. 3–4.

C. Claim Construction

In an *inter partes* review, claim terms in an unexpired patent are given their broadest reasonable construction in light of the specification of the patent in which they appear. 37 C.F.R. § 42.100(b); *In re Cuozzo Speed Techs., LLC*, No. 2014-1301, 2015 WL 4097949, at *5–7 (Fed. Cir. July 8, 2015) (confirming that the broadest reasonable construction standard was properly adopted by PTO regulation). In determining the broadest reasonable construction, we presume that claim terms carry their ordinary and customary meaning. *See In re Translogic Tech., Inc.*, 504 F.3d 1249, 1257 (Fed. Cir. 2007). This presumption may be rebutted when a patentee,

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[4] U.S. Patent No. 3,634,944 (Jan. 18, 1972) (Ex. 1004).

[5] EP Patent Publication No. 0374709 A2 (Published June 27, 1990) (Ex. 1006).

acting as a lexicographer, sets forth an alternate definition of a term in the specification with reasonable clarity, deliberateness, and precision. *In re Paulsen*, 30 F.3d 1475, 1480 (Fed. Cir. 1994).

Petitioner proposes constructions for numerous claim terms of the ’329 patent. Pet. 14–19. For purposes of this Decision, only the term “by injection of an inert gas” requires discussion.

Claim 1 requires removing the gelatinous reaction mixture “by injection of an inert gas.” Ex. 1001, 14:39–40. Petitioner argues that this phrase should be given its plain and ordinary meaning, which is broad enough to encompass indirectly *facilitating* the discharge or removal of the gelatinous reaction mixture by, for example, injecting an inert gas to drive a piston. Pet. 19, 24 n.2.

Patent Owner contends that Petitioner’s construction is unreasonably broad because the claim term “by” identifies “the agent performing the action,” i.e., an inert gas, and the Specification consistently discloses the use of gas pressure (or liquid pressure) to remove the gelatinous product, with no mention of a piston. Prelim. Resp. 12–17 (citing Ex. 1001, 6:66–7:6 (“In order to discharge the polymer gel from the tubular reactor; an inert gas is injected onto the polymer gel at the top of the tubular reactor . . . .”), 8:63–65; 9:17–19; 9:62–63; 10:18–19; 10:53–57; 11:16–19; 11:47–49; 12:5–7; 12:33–37; 12:64–66; 13:26–28; 13:56–58; 14:21–23 (describing the use of an inert gas (or liquid) to expel the gelatinous mixture, with no reference to a piston)). Patent Owner further asserts that the Specification explicitly distinguishes removal of the polymer gel “by the injection of an inert gas” from removal by use of a piston. *Id.* at 16.

We agree with Patent Owner that the term "by injection of an inert gas" cannot reasonably be interpreted to encompass the use of a piston driven by inert gas. As noted by Patent Owner, the '329 patent repeatedly, consistently, and exclusively discusses the use of an inert gas (or liquid) to remove polymer gels from a reactor—without reference to a piston. *See In re Abbott Diabetes Care Inc.*, 696 F.3d 1142, 1150 (Fed. Cir. 2012). Moreover, the '329 patent Specification, in discussing the disclosure of Japanese Application JP-A-93/57181 (Ex. 1014, "JP '181"), distinguishes removal "by injection of an inert gas" from removal by use of a piston. Ex. 1001, 1:23–35.

Accordingly, we construe the term "by injection of an inert gas" to require the inert gas to act directly upon the gelatinous reaction mixture.

II. ANALYSIS OF GROUNDS

A. Obviousness of Claim 1–3 and 5–7 over the '944 Patent and the '597 Patent

Petitioner asserts that claims 1–3 and 5–7 are unpatentable under 35 U.S.C. § 103 as obvious over the '944 patent and the '597 patent. Pet. 4. In support of this argument, Petitioner provides detailed analysis showing where each claim limitation is allegedly disclosed or suggested in the recited reference. Pet. 44–51. Petitioner also provides the declaration testimony of Dr. Benny Freeman. Ex. 1007.

1. The '944 Patent

The '944 patent is directed to the production and drying of polyacrylamide gels. Ex. 1004, 1:4–9. "The immediate product of the polymerization is a clear hydrous gel" that is "intensely adhesive" and "so viscous as to be self-supporting." *Id.* at 1:22–26. Figure 1A of the '944 patent is depicted below:

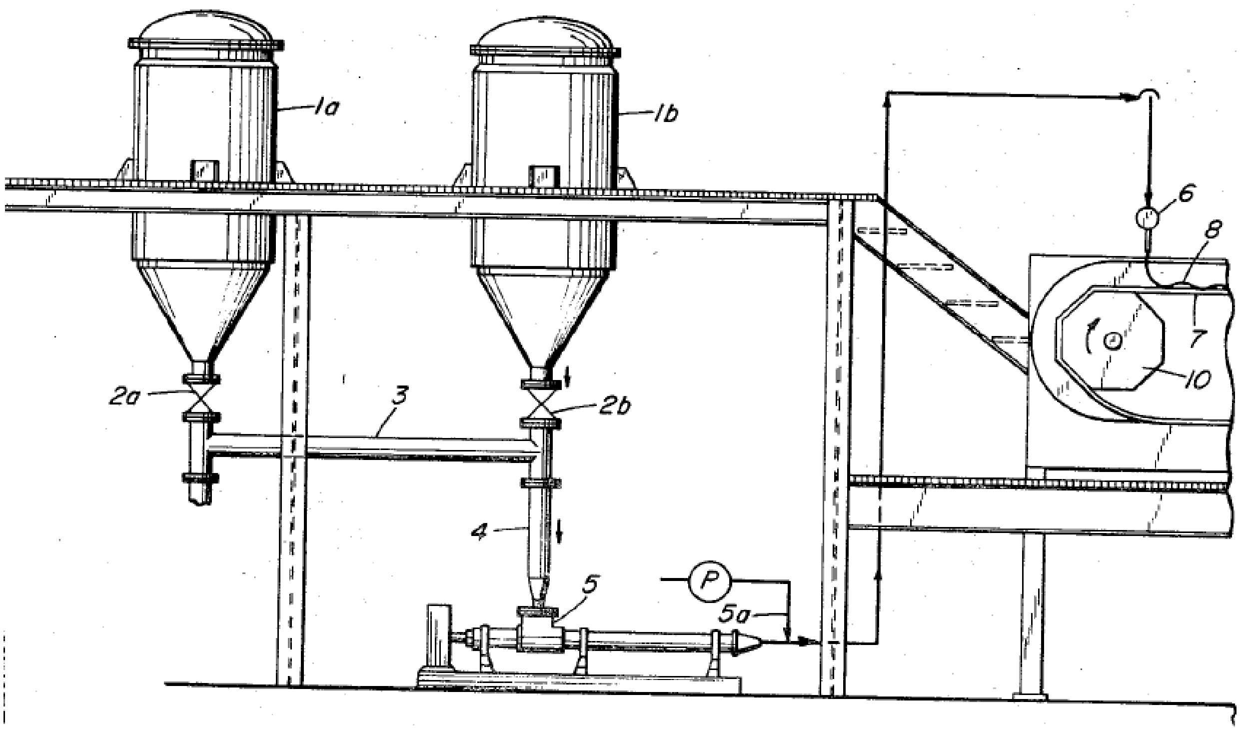


FIG. 1A

Figure 1A of the ’944 patent “is an elevation showing schematically the reactors in which the acrylamide polymer gel is formed and the end of the first travelling belt on which the gel from the reactors is discharged.” *Id.* at 3:7–10. In this figure, tubular polymerization chambers 1a and 1b have a conical taper and supply an acrylamide polymer gel, through valves 2a and 2b and pipes 3 and 4, to screw pump 5. *Id.* at 3:23–28. The gel is then supplied under pressure to header 6 and extruded on steel belt 7 in the form of cords 8. *Id.* at 3:28–31.

2. The ’597 Patent

The ’597 patent is directed to a process for polymerizing acrylamide in an aqueous solution using polymerization catalysts, such as azobisisobutyronitrile. Ex. 1002, 1:2–3, 1:22–30, 2:58–65. The ’597 patent discloses that "[a]ny conventional reaction vessel may be used" in the process, but tubular-shaped reactors having a bottom product outlet for discharging the gel-like polymer are preferred. *Id.* at 2:70–3:2. After polymerizing the monomers in the reaction chamber, nitrogen gas is injected into the reactor to discharge the gel into an extruder. *Id.* at 3:70–75.

In Example 1 of the '597 patent, the reaction vessel is eight centimeters in diameter, fifty centimeters in height, and has an outlet two centimeters in diameter at the bottom of the vessel. *Id.* at 3:60–62. In Example 2, the reaction vessel is sixty centimeters in diameter, two-hundred centimeters in height, and has an outlet ten centimeters in diameter at the bottom of the vessel. *Id.* at 4:34–36.

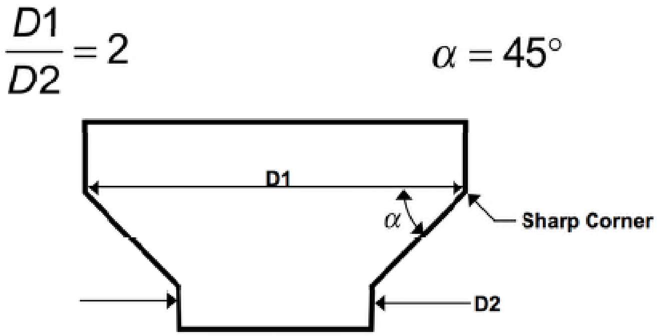
3. *Analysis*

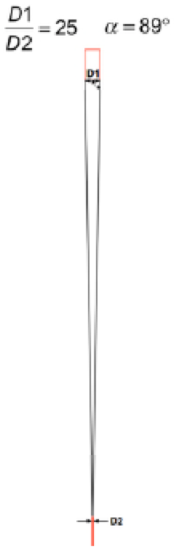
Petitioner asserts that in combination the '944 patent and the '597 patent disclose or suggest every element of claims 1–3 and 5–7 of the '329 patent. For example, with respect to claims 1 and 2, Petitioner asserts that both patents disclose the polymerization of water soluble, monoethylenically unsaturated monomers (acrylamide) in an aqueous solution in the presence of a polymerization initiator. Pet. 44–45, 48–49. Petitioner further asserts that the '944 patent discloses producing polyacrylamide in a tubular reaction chamber that has a conical taper and the '597 patent discloses successfully removing the same type of polyacrylamide gel using inert gas pressure and D1:D2 ratios within the 2:1 to 25:1 range set forth in claim 1. Pet. 44–45, 50 (citing Ex. 1002, 3:60–73, 4:34–45).

Although neither reference explicitly discloses an angle α for the conical taper, Dr. Freeman testifies that one of ordinary skill in the art would have selected, using routine optimization, a taper angle for the '944 patent that is greater than 45 degrees and less than 90 degrees (claim 1), as well as a taper angle that is greater than 65 degrees and less than 85 degrees (claim 2). Pet. 47 (citing Ex. 1007 ¶ 238); *see id.* at 29–32 (citing Ex. 1007 ¶¶ 36, 39, 42, 161, 165, 183–185, 188, 192, 195–196).

Dr. Freeman asserts that a person of ordinary skill in the art would have used an angle greater than 45 degrees, and preferably above 65 degrees as recited in dependent claim 2, to minimize "dead zones," or localized zones of low flow, usually near corners, caused by flow disturbances. Ex. 1007 ¶¶ 36, 40. According to Dr. Freeman, "dead zone reduction was and is one of the most important factors considered in design of reactors." *Id.* ¶ 39. Dr. Freeman further asserts that one of ordinary skill in the art would have used an angle that is less than 85 degrees (as recited in claim 2) because a reactor with a taper that is between 85 and 90 degrees would "need to be excessively tall" and would not provide the added benefit of using a taper. *Id.* ¶¶ 192, 238.

Indeed, Dr. Freeman testifies that, given the broad scope of reactors encompassed by the claimed D1:D2 ratios and α angles, and given the need to reduce "dead zones" and to maximize the force of the inert gas pressure pushing the polymer gel towards the outlet, one of ordinary skill in the art *would not* have selected a reactor that is outside the bounds of claim 1. *Id.* ¶¶ 183–185. In support of this argument, Dr. Freeman provides the following drawings, allegedly drawn to scale:





Id. ¶ 183. At the one extreme of D1:D2 of 2 and α of 45 degrees (figure on the left), Dr. Freeman contends that the reactor is "dish-like" and would not reduce dead zones or provide the optimal benefits of tapering. *Id.* ¶¶ 182, 184, 187 (noting that at a conical angle of 45 degrees, the downward force on the gel "is equal to the component of force pushing the gel-like mixture towards the wall of the reactor, thereby increasing the friction of this gel-like mixture with the wall, impeding its discharge"). At the other extreme of D1:D2 of 25 and α of 89 degrees (the figure on the right), Dr. Freeman contends that the reactor is "needle-like" with a conical section that is 27.5 times longer than the diameter of the reactor. *Id.* ¶¶ 185–186.

At this stage of the proceeding, we credit Dr. Freeman's testimony that a person of ordinary skill in the art would have selected, using routine optimization, a taper angle for the '944 patent reactor that is within the scope of claim 1 (>45° and <90°) and claim 2 (from 65° to 85°).

Petitioner also presents evidence that each element of dependent claims 3 and 5–7 is disclosed in, or rendered obvious by, the '944 and '597 patents. For example, Petitioner asserts that both references disclose polymerizing acrylamide (claim 3) using azo initiators, such as azobisisobutyronitrile (claim 5). Pet. 51. Petitioner also presents evidence, supported by the testimony of Dr. Freeman, that one of ordinary skill in the art would have routinely optimized, based on various conformational and physical factors, the applied inert gas pressure to be within the levels recited in claim 6 (2 to 65 bar) and claim 7 (4 to 25 bar). *Id.* at 47–48 (citing Ex. 1007 ¶¶ 240–243 (asserting that, once the discharge orifice and reaction mix viscosity are determined, "[s]electing a pressure within the '329 claims is virtually automatic," and would be "a matter of routine optimization")).

As for the rationale for combining the references, Petitioner asserts that combining the disclosed features of the '944 and '597 patents would have been obvious to one of ordinary skill in the art because it involves "merely the combination of known elements according to known methods to yield predic[t]able results." *Id.* at 47.

Patent Owner argues that the challenged claims would not have been obvious over the '944 and '597 patents because the prior art teaches away from using an inert gas to remove polymer gels. Prelim. Resp. 44–45. In particular, Patent Owner contends JP '181 discloses that removing polymerized gel from a reactor vessel using inert gas pressure "simply does not work." *Id.* We are not persuaded by this argument because the '597 patent discloses successfully removing polymer gels from a tubular reactor, and we are directed to no disclosure in JP '181 indicating that inert gas pressure "does not work." *Id.* at 45 (citing Ex. 1014 ¶ 3); Ex. 1002, 2:70–3:2, 3:70–73 (disclosing the successful removal of polyacrylamide gels from a tubular reaction vessel using inert gas pressure). Indeed, although the recited portion of JP '181 does disclose that the use of inert gas pressure presents certain "difficulties," such as gas escaping between gaps along the reactor walls, it also discloses that this method was known in the art and conventional. Ex. 1014 ¶¶ 2–3; *In re Gurley*, 27 F.3d 551, 553 (Fed. Cir. 1994) (noting that a reference that allegedly taught away from the use of epoxy also taught that epoxy is usable and had been used for the same purpose as set forth in the disputed patent).

Patent Owner also argues that, due to "the problems identified in the prior art regarding the use of inert gas to achieve a *complete discharge* of viscous and sticky polymer gels from polymerization reactors and the express solution to this problem provided in the prior art, namely the use of a piston," one of ordinary skill in the art would not "reasonably expect to successfully remove polymer gel from a reactor" using inert gas pressure. Prelim. Resp. 27–28 (emphasis added); *see also id.* at 26 (asserting that EP '709 discloses "it was difficult to achieve a complete and efficient discharge with gas pressure"). We are not persuaded by this argument because Patent Owner has not explained why the challenged claims, as properly construed, require any particular efficiency level for the removal of the polymer gel, much less "complete removal" of the gel from the reaction vessel. *Id.* at 27, 29, 34–35.

Patent Owner further argues that neither the '597 patent nor the '944 patent discloses a D1:D2 ratio for a tubular reactor *with a conical taper*. Prelim. Resp. 50–51. Patent Owner's argument, however, does not address the specific combination of prior art elements asserted by Petitioner—Petitioner does not argue that either reference explicitly discloses a D1:D2 ratio for a conical taper but, instead, that one of ordinary skill in the art would select the D1:D2 ratio of the '597 patent for use with the conical taper of the ’944 patent. Pet. 47 (“Therefore, it would have been obvious to use the ’597 patent’s inert gas pressure and D1:D2 ratio of 6:1 to discharge the gel from the funnel cone reactors disclosed in the ’944 patent.”); Ex. 1007 ¶¶ 235–236.

Patent Owner also contends that Petitioner fails to establish that taper angles were a known variable that would have been optimized by one of ordinary skill in the art when designing a polymerization reactor. Prelim. Resp. 41 (citing *In re Antonie*, 559 F.2d 618, 629 (CCPA 1977)). At this stage of the proceeding, we are not persuaded by this argument because, as discussed above, Dr. Freeman provides detailed testimony explaining the various design considerations that would have been addressed by one of ordinary skill in the art when selecting an angle for the ’944 patent taper, including avoiding “dead zones” and maximizing the amount of pressure that is exerted towards the reactor outlet, as opposed to the reactor walls. *See* Pet. 29–33, 47–48; Ex. 1007 ¶¶ 39, 167, 186–188.

Patent Owner further argues that, even if taper angles were a known variable for optimization, Petitioner’s argument that the claimed taper angles could be arrived at through routine optimization is based on references related to “bulk solid particles in a hopper,” which Dr. Freeman has not shown to be “relevant to sticky, viscous polymer gels.” Prelim. Resp. 41–42. At this stage of the proceeding, we are not persuaded by this argument because, although Dr. Freeman supports his analysis with references discussing the gravity feed of bulk solids, he also explains why dead zones are affected by taper angle and provides analysis of the forces, and the direction of those forces, that would be applied on the viscous polymer gels disclosed in the ’329 patent at various taper angles. *See, e.g.*, Ex. 1007 ¶¶ 182, 187, 194.

4. Conclusion

On the current record, Petitioner identifies where each claim element is allegedly disclosed in, or suggested by, the ’944 and ’597 patents. Petitioner also articulates a credible rationale to support the proposed combination of the prior art references. *See KSR Int’l v. Teleflex, Inc.*, 550 U.S. 398, 416 (2007) (“The combination of familiar elements according to known methods is likely to be obvious when it does no more than yield predictable results.”); *see also id.* at 417 (“[I]f a technique has been used to improve one device, and a person of ordinary skill in the art would recognize that it would improve similar devices in the same way, using the technique is obvious unless its actual application is beyond his or her skill.”). We are persuaded, therefore, that Petitioner has demonstrated a reasonable likelihood that claims 1–3 and 5–7 would have been obvious over the ’944 patent and the ’597 patent.

B. Obviousness of Claims 4 and 5 over the ’944 Patent, the ’597 Patent, GB ’028, the ’460 Patent, and EP ’709

Claim 4 depends from claim 1 and further requires “wherein the water-soluble monoethylenically unsaturated monomers are polymerized with from 0.001 to 5% by weight, based on the total monomers employed in the polymerization, of at least one crosslinker.” Ex. 1001, 14:49–53. Petitioner contends that EP ’709 discloses polymerizing acrylamide together with at least one cross-linker, “typically in amount of 0.01-1 mol%, based on the total quantity of monomers.” Pet. 57 (citing Ex. 1006, 3:1–3).

In support of Petitioner’s argument, Dr. Freeman testifies that 0.01–1 mol % is within the 0.001 to 5 weight % range of claim 4. Ex. 1007 ¶¶ 262,

275. Dr. Freeman further testifies that crosslinkers were known to cause the polymer structure to become more networked and structured, "creating valuable superabsorbent materials." Ex. 1007 ¶ 277. Patent Owner does not specifically address Petitioner's arguments with respect to claim 4.

On this record, we are persuaded that Petitioner has demonstrated a reasonable likelihood that claim 4 would have been obvious over the '944 patent, the '597 patent, and EP '709. Petitioner does not explain, however, why GB '028 and the '460 patent provide any additional disclosure in support of this proposed combination than that provided by the '944 and '597 patents. *See* Pet. 57–58. Accordingly, we exercise our discretion and do not institute *inter partes* review with respect to these additional references. *See* 35 U.S.C. § 314(a); 37 C.F.R. § 42.108(a); *see also* 37 C.F.R. § 42.1(b) (proceedings before the Board are to be construed to "secure the just, speedy, and inexpensive resolution of every proceeding").

Petitioner also asserts that claim 5 is obvious over the '597 patent, the '944 patent, GB '028, the '460 patent, and EP '709. Pet. 52–54. Petitioner does not explain, however, why this set of references is stronger than the combination of the '944 patent and the '597 patent discussed above with respect to claim 5. Accordingly, we apply our discretion and decline to institute review of claim 5 over the '944 patent, the '597 patent, GB '028, the '460 patent, and EP '709.

C. The Additional Asserted Grounds

Petitioner asserts that claims 1–7 also would have been obvious over various additional combinations of GB '028, the '597 patent, the '460 patent, the '944 patent, and EP '709. Pet. 3–4. Petitioner does not explain why any of these asserted grounds is stronger than the ground based on the

’944 patent and the ’597 patent (for claims 1–3 and 5–7) or the ground based on the ’944 patent, the ’597 patent, and EP ’709 (for claim 4) discussed above. As our institution of review includes each challenged claim, we exercise our discretion and decline to institute *inter partes* review on the additional asserted grounds incorporating GB ’028, the ’597 patent, the ’460 patent, the ’944 patent, and EP ’709.

IV. ORDER

For the foregoing reasons, it is

ORDERED that pursuant to 35 U.S.C. § 314 an *inter partes* review of the ’329 patent is hereby instituted on the following grounds:

1. Whether claims 1–3 and 5–7 would have been obvious under 35 U.S.C. § 103 over the ’944 patent and the ’597 patent; and

2. Whether claim 4 would have been obvious under 35 U.S.C. § 103 over the ’944 patent, the ’597 patent, and EP ’709;

FURTHER ORDERED that the trial is limited to the grounds identified above and no other grounds are authorized; and

FURTHER ORDERED that pursuant to 35 U.S.C. § 314(a), *inter partes* review of the ’329 patent is hereby instituted commencing on the entry date of this Order, and pursuant to 35 U.S.C. § 314(c) and 37 C.F.R. § 42.4, notice is hereby given of the institution of trial.

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F.3d 1331, 1337 (Fed. Cir. 2015); *see also In re Cyclobenzaprine*, 676 F.3d at 1082-83 ("Evidence that others were 'going in different ways' is strong evidence that the inventor's way would not have been obvious."). There is strong, objective, hindsight-free evidence of nonobviousness.

B. The '329 Invention Produced Unexpected Results Over the Closest Prior Art

Because "that which would have been surprising to a person of ordinary skill in a particular art would not have been obvious," unexpected properties or results of an invention refute a contention that it was obvious. *In re Soni*, 54 F.3d 746, 750 (Fed. Cir. 1995). As described above, prior to the invention of the '329 patent, conical tapers and inert gas had never been used to remove polymer gels from reactors. For that reason, and because no art even suggested conical tapers and inert gas could be used in that way, test results showing pairing conical tapers and inert gas at the claimed ranges was able to remove polymer gel from a reactor with little or no residue were highly unexpected and surprising. Ex. 2016 ¶ 206. The inventors experiments unequivocally revealed the claimed process could be applied to a wide range of polymer gels with different properties (e.g., crosslinked and not crosslinked) and all were able to be removed from the reactor with little or no residue. *Id.* ¶¶ 212-16. Compared to the closest prior art of the EP '709, as well as the '944 patent on which Petitioner relies (which, as noted above, display either

Jan. 8, 1974 TAKEHIKO FUJIMOTO ET AL **3,784,597**

PROCESS FOR PRODUCING A PARTIALLY HYDROLYZED ACRYLAMIDE POLYMER

Filed Dec. 16, 1971

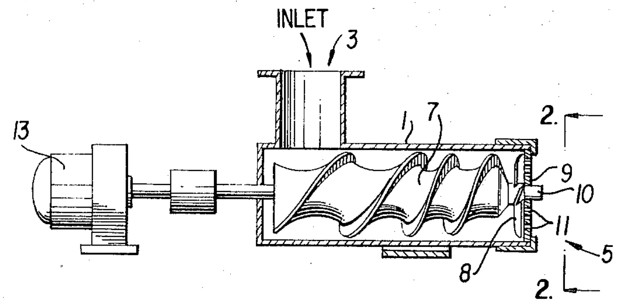


FIG. 1

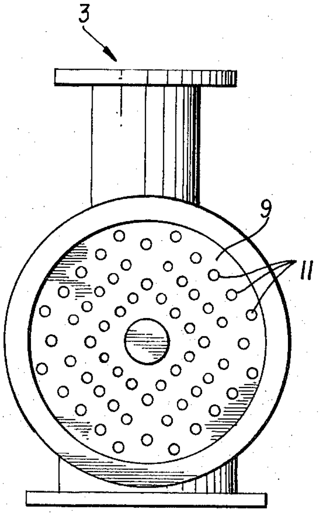
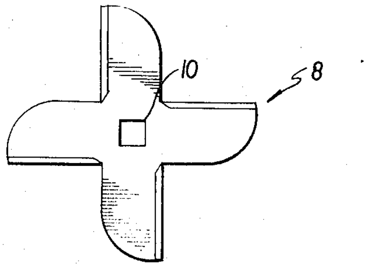


FIG. 2

FIG. 3



United States Patent Office

3,784,597
Patented Jan. 8, 1974

3,784,597

PROCESS FOR PRODUCING A PARTIALLY HYDROLYZED ACRYLAMIDE POLYMER

Takehiko Fujimoto, Masashi Okada, and Shigeo Inoue, Kyoto, Japan, assignors to Sanyo Chemical Industries, Ltd., Kyoto, Japan

Filed Dec. 16, 1971, Ser. No. 208,875
Claims priority, application Japan, Dec. 19, 1970, 45/114,562
Int. Cl. C08f *3/90, 27/14*
U.S. Cl. 260—80.3 N **8 Claims**

ABSTRACT OF THE DISCLOSURE

Acrylamide polymers and copolymers are partially hydrolyzed in an extruder from high concentration gel-like aqueous medium.

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to a partially hydrolyzed acrylamide polymer. As used herein, the term "acrylamide polymer" is intended to refer to either acrylamide homopolymers or acrylamide containing copolymers. More particularly, this invention relates to a process for hydrolyzing an acrylamide polymer in a gel-like aqueous medium of high concentration. As used herein the term "aqueous medium" is intended to refer to aqueous solutions, emulsions or dispersions.

Description of prior art

Partially hydrolyzed acrylamide polymers are among the more useful of the flocculants, for the flocculation of aqueous suspensions, such as in the treatment of industrial or residential sewage, or the like.

Partially hydrolyzed acrylamide polymers are of particular interest as aqueous suspension flocculating agents, which is finding increasing demand for use in the treatment of industrial sewage or the like.

Conventionally, acrylamide polymers are obtained by polymerizing the acrylamide, alone or with other monomers in very dilute aqueous solutions. The resulting polymer in the dilute aqueous solution is partially hydrolyzed with sodium hydroxide, sodium bicarbonate or sodium carbonate.

Very dilute aqueous solutions are necessary in the conventional hydrolysis process because high concentrations of acrylamide solutions, that is, solutions containing more than 10% by weight polymer, are usually very viscous, sticky, gel-like solutions.

While it has been considered to use other solvents instead of water to form the partially hydrolyzed acrylamide polymer solutions, only water has been shown to be a suitable medium for the hydrolysis reaction.

The difficulty with using such dilute solutions are that (a) they are degradable and perishable during storage, (b) they are inconvenient or difficult to transport and handle, since the bulk of the solution consists of water.

Although some attempts have been made to hydrolyze the high concentration gel-like solution of the acrylamide polymers, they have been largely unsuccessful, since it has been almost impossible to homogeneously distribute the base material through the thick gel solution, by ordinary stirring or mixing techniques. If more vigorous agitation is used, it can result in significant decomposition of the hydrolyzed polymer.

One reported attempt at hydrolysis of the gel state solution was to cut the solution into small particles of less than 0.5 inch in diameter and then to contact these particles with an alkaline solution. This technique has not proven to be successful, however, largely because the degree of hydrolysis is dependent upon the distance of the polymer molecule from the particle surface.

Accordingly, a need continues to exist for a technique of hydrolyzing acrylamide polymer in high concentrations.

SUMMARY OF THE INVENTION

Accordingly, it is one object of this invention to provide a process for partially hydrolyzing acrylamide polymers in a high concentration, without causing chain degradation.

It is another object of this invention to provide a process for partially hydrolyzing acrylamide polymers from an aqueous gel state.

These and other objects of this invention, as will hereinafter become more readily apparent, have been attained by homogeneously mixing a high concentration gel-like acrylamide polymer aqueous medium with an alkaline material, in an extruder.

BRIEF DESCRIPTION OF THE DRAWINGS

In describing this invention, reference shall be made to the accompanying drawings, in which:

FIG. 1 is a partial cross-sectional view of an extruder which is suitable for the mixing operation of this invention;

FIG. 2 is a front view of the extruder head of said extruder; and

FIG. 3 is a cutting blade used at the outlet of the extruder.

DETAILED DESCRIPTION OF THE INVENTION

The acrylamide polymer medium used in this invention is an acrylamide polymer solution (or emulsion or dispersion) in an amount of at least 10% by weight, in water. Preferably, the concentration is between 15–50% by weight. Less than 10% by weight will cause the medium to have too low a viscosity for successful mixing in the extruder. Within the 15–50% by weight range, however, a gel-like state will be formed which can easily be handled by the extruder.

The acrylamide polymer used in this invention is preferably the homopolymer of acrylamide or a hydrophilic copolymer of acrylamide with less than 50%, and preferably less than 30% by weight of one or more suitable comonomers, such as: methacrylamide, acrylic acid, acrylonitrile, acrylic esters (e.g., methyl, ethyl, propyl and butyl acrylates), methacrylic esters (e.g., methyl, ethyl, propyl and butylmethacrylates), styrene, vinyl alkyl ethers, vinyl chloride, vinyl acetate and vinylidene chloride.

Suitable acrylamide polymers are those having a viscosity of at least 1000 cps. (usually, 1,000–15,000 cps. and preferably 3,000–10,000 cps.) as determined by a Brookfield viscometer, 12 rounds per minute, Roter #3, in 1% by weight aqueous solution at 30° C.

The acrylamide polymer containing medium is produced by polymerizing acrylamide with or without other comonomers, in an aqueous medium containing at least 10% by weight, and preferably 15–50% by weight of the monomer. Polymerization can be effected by any conventional technique such as by free radical polymerization using such catalysts as ammonium persulfate, potassium persulfate, sodium persulfate or azobisisobutyronitrile. Conventional reducing agents, such as sodium sulfite, sodium hydrogen sulfite, sodium thiosulfate, hydroxyl amine, hydrazine or ferrous ion, may be used therewith. Polymerization may be effected at temperatures of 20°–130° C. in an inert atmosphere. Any conventional reaction vessel may be used, preferably those having a bottom product outlet for discharging the gel-like polymer containing medium, usually at elevated pressures. Also, tubular-shaped reactors are quite suitable for this purpose.

The gel-like acrylamide polymer containing medium is homogeneously mixed with an alkaline material in an extruder at temperatures of 10°–150° C.

Any extruder which can provide the functions of continuous mixing and continuous extrusion can be used for this invention.

One suitable extruder is shown in FIG. 1, showing a cylindrical housing **1** having an inlet **3** at one end, and an outlet **5** at the opposite end. A rotating screw agitator **7** is provided within the housing which not only homogeneously mixes the materials charged into the extruder but also transfers them from the inlet **3** to the outlet **5**. As shown in FIGS. 1 and 2, the outlet has a nozzle plate **9** containing nozzle apertures **11** of diameter *d*. A cutting blade **8** shown in FIG. 3 is preferably attached to the shaft **10** of the rotating screw **7**, inside of the outlet **5** of the extruder, which acts to cut the product into particles. The rotating screw is driven by motor **13**.

The acrylamide polymer containing medium, preferably the aqueous solution, in its gel-like state and the alkaline material are introduced into the extruder through inlet **3** and are mixed and carried through the extruder to the outlet nozzle apertures **11** where they are extruded into a particle form.

Suitable alkaline materials which can be used for the hydrolysis are the alkali metal hydroxides (e.g., sodium and potassium hydroxides), and alkali metal salts of weak acids (e.g., sodium and potassium carbonates, bicarbonates, phosphates and carboxylates). The quantity of alkaline material is not critical and may vary over a wide range. It is preferable, however, to use at least an equivalent amount based on the number of amide groups to be hydrolyzed. The alkaline compound may be used in the form of an aqueous solution.

The product of this invention is a partially hydrolyzed acrylamide polymer, wherein the percentage of hydrolysis is generally 1–70% of all of the amide groups (preferably 5–50%).

This invention has the significant advantages that high concentrations of the acrylamide polymers can be hydrolyzed, and the resulting product will have a uniform degree of hydrolysis with very little degradation of the polymer carbon chain; that is, the product will have almost the same viscosity as the product obtained in a dilute aqueous solution by conventional techniques, and it will function as good flocculants.

Having generally described the invention, a further understanding can be attained by reference to the following examples which are presented as illustrative of this invention only, and it will be understood that the invention is not limited thereto unless otherwise specifically specified. The viscosities in the examples were determined in 1% by weight aqueous solution of acrylamide polymers or hydrolytes thereof. All parts are "by weight" unless otherwise specified.

EXAMPLE 1

Into a reaction vessel 8 cm. in diameter and 50 cm. in height having an outlet 2 cm. in diameter at the bottom of the vessel, was charged 250 parts of acrylamide and 750 parts of water. The mixture was heated at 20° C. under a nitrogen atmosphere. To the mixture was added 0.05 part of ammonium persulfate and 0.024 part of sodium hydrogen sulfite while stirring. The mixture was then maintained at 50–80° C. for 2 hours. There was obtained a gel-like polymer solution whose polymer had a viscosity of 1,200 cps.

The resulting gel-like polymer solution was discharged from the vessel through the outlet by use of nitrogen gas pressure, and mixed homogeneously with 56 parts of sodium hydroxide powder at 65° C. in an extruder as shown in the drawings, which comprises a cylinder 6 cm. in diameter and 15 cm. in length, having an inlet and an outlet, a rotating screw equipped therein in parallel, a blade attached to the screw at the outlet side, and a nozzle plate having many nozzle apertures (40% of opening) of 2 mm. in diameter. The retention time of the mixture in the extruder was about 30 seconds. There was obtained a gel-like hydrolyte solution whose polymer had a degree of hydrolysis of 38% by mole.

The resulting hydrolyte was compared with that produced by conventional methods, as to viscosity and flocculating properties. The results are provided in Table I, which show that no chain degradation occurred in the hydrolyte of this invention.

TABLE I

Hydrolyte	Degree of hydrolysis, percent	Viscosity, cps.	Flocculating property
Hydrolyte of Example 1	38	2,300	Good.
Conventional hydrolyte:			
(A)	38	2,300	Do.
(B)	38	1,000	Poor.

NOTE.—Flocculating was observed by adding, respectively, 1 p.p.m. (active component) of hydrolyzed polymers to 0.5% by weight $Mg(OH)_2$ aqueous suspensions.

The conventional hydrolyte (A) in Table I was produced by diluting the gel-like polyacrylamide solution, obtained as in Example 1, with water to make a 3% by weight solution of the polymer, and hydrolyzing the resulting solution at 70° C. for 1 hour under mild agitation. The conventional hydrolyte (B) in Table I was produced by hydrolyzing a mass of the gel-like polyacrylamide solution (obtained as in Example 1) at room temperature under vigorous agitation.

EXAMPLE 2

Into a reaction vessel, 60 cm. in diameter and 200 cm. in height, having an outlet of 10 cm. in diameter at the bottom of the vessel, was charged 250 parts of acrylamide and 750 parts of water. The mixture was heated at 50° C. under a nitrogen atmosphere and to the mixture was added 0.025 part of ammonium persulfate while stirring. The mixture was then maintained at 90°–120° C. for 2 hours. There was obtained a gel-like polymer solution whose polymer had a viscosity of about 2,400 cps.

The resulting gel-like solution was discharged from the vessel through the outlet by applying nitrogen gas pressure and was introduced at 90° C. into the same type of extruder as in Example 1, except that the cylinder was 15 cm. in diameter and 60 cm. long, and the nozzle plate had nozzle apertures of 6 mm. in diameter, at about 30% of opening percentage. To the gel-like solution was added continuously 70 parts of 50% NaOH aqueous solution at the inlet of the extruder.

There was obtained a gel-like hydrolyte solution whose polymer had a degree of hydrolysis of about 25% by mole and viscosity of about 6,400 cps.

EXAMPLE 3

Example 2 was repeated, except using 225 parts of acrylamide, 25 parts of ethyl acrylate, 750 parts of water, 0.025 part of ammonium persulfate, and 40 parts of 50% NaOH aqueous solution. The copolymer in gel-like solution had a viscosity of about 1,300 cps. The final hydrolyte in the gel-like solution had a degree of hydrolysis of about 15% by mole and a viscosity of about 3,400 cps.

EXAMPLE 4

Example 2 was repeated, except using 225 parts of acrylamide, 25 parts of methyl methacrylate, 750 parts of water, 0.025 part of ammonium persulfate and 40 parts of 50% NaOH aqueous solution. The copolymer in gel-like solution had a viscosity of about 1,500 cps. The final hydrolyte in the gel-like solution had a degree of hydrolysis of about 15% by mole and a viscosity of about 3,600 cps.

Having now fully described the invention, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without departing from the spirit or scope of the invention.

What is claimed as new and desired to be secured by Letters Patent is:

1. In a process for partially hydrolyzing an acrylamide polymer the improvement comprising

forming a gel-like aqueous state containing at least 10% of acrylamide polymer wherein said acrylamide polymer contains less than 50% of a comonomer and has a viscosity of at least 1,000 cps. in a 1% by weight aqueous solution,

admixing said gel with an alkaline material while maintaining a gel-like state,

extruding said gel-like state acrylamide polymer so as to partially hydrolyze said polymer to a degree of 5–50%.

2. The process according to claim **1** in which the acrylamide polymer in the gel-like aqueous solution is a solution of an acrylamide polymer which has been produced by polymerizing acrylamide or a mixture of acrylamide with at least one comonomers in an aqueous solution of at least 10% by weight.

3. The process according to claim **1** in which the alkaline compound is an alkali metal hydroxide or an alkali metal salt of a weak acid.

4. The process according to claim **1** in which the acrylamide polymer in a gel-like aqueous solution form and the alkaline material is continuously mixed and continuously extruded.

5. The process according to claim **1** which comprises feeding the gel-like aqueous solution and the alkaline material into an extruder inlet, longitudinally transferring while simultaneously homogeneously mixing said solution and said alkaline material, and extruding said mixed product through an extrusion outlet.

6. The process according to claim **5**, wherein the product is extruded in small pieces.

7. The process according to claim **1** in which the alkaline material is used in approximately an equivalent amount based on the amide groups in the acrylamide polymer to be hydrolyzed.

8. The process according to claim **1** in which the hydrolysis is carried out at temperatures of 10°–150° C.

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U.S. Cl. X.R.

260—85.7, 87.5 R, 87.7, 88.1 PN, 89.7 S; 264—140

United States Patent

Zonis et al.

[15] **3,634,944**

[45] **Jan. 18, 1972**

[54] **DRYING OF STICKY THERMOSENSITIVE HYDROUS GELS**

[72] Inventors: **Meyer Louis Zonis**, Livingston; **Girish Chandulal Shah**, Wayne, both of N.J.; **Kenneth Worden Saunders**, Darien; **Michael Niall Desmond O'Connor**, Norwalk, both of Conn.

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[22] Filed: **June 1, 1970**

[21] Appl. No.: **41,807**

[52] **U.S. Cl.** **34/12,** 34/18
[51] **Int. Cl.** **F26b 7/00**
[58] **Field of Search** 34/9, 12, 18

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Primary Examiner—John J. Camby
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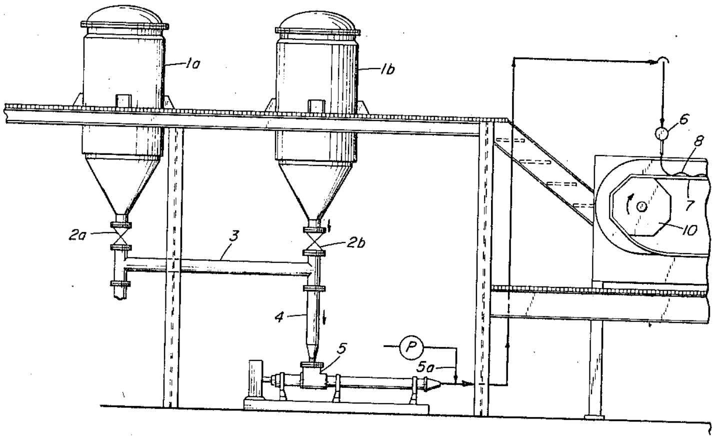
[57] **ABSTRACT**

A sticky water-soluble high-viscosity polyacrylamide hydrous gel is dried without significant decrease to its water-solubility by extruding the gel as paralleled cords upon a fast-travelling belt having a nonadherent surface and heating the cords with high-temperature gas until the surface of the cords is non-tacky, and then discharging the cords upon a slowly travelling belt and completing the drying with low-temperature gas.

12 Claims, 4 Drawing Figures



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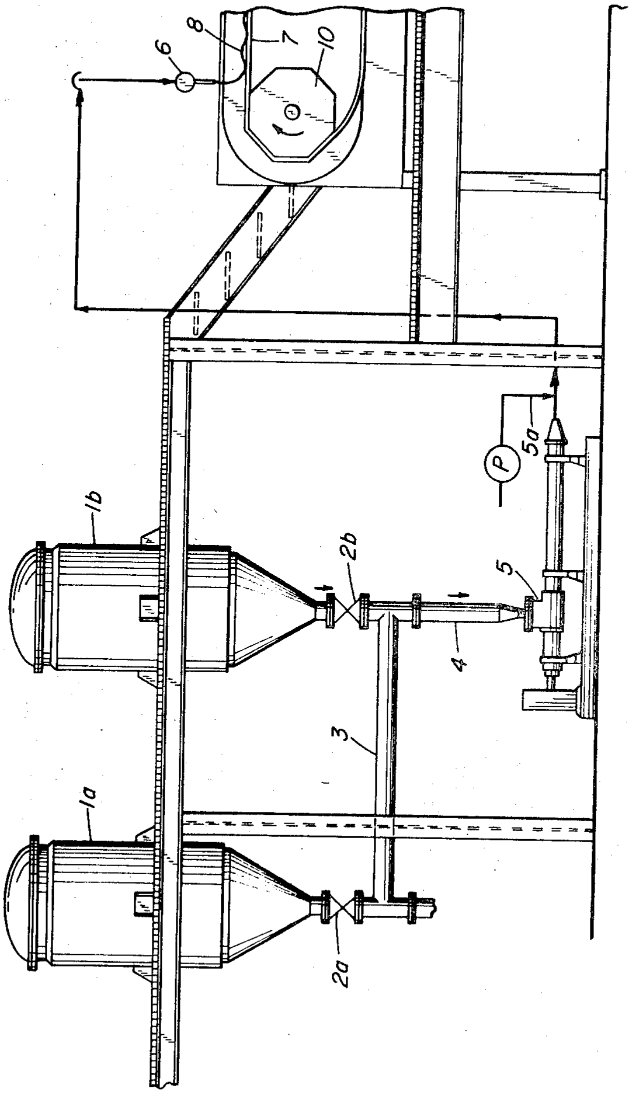

FIG. 1A

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PATENTED JAN 18 1972

3,634,944

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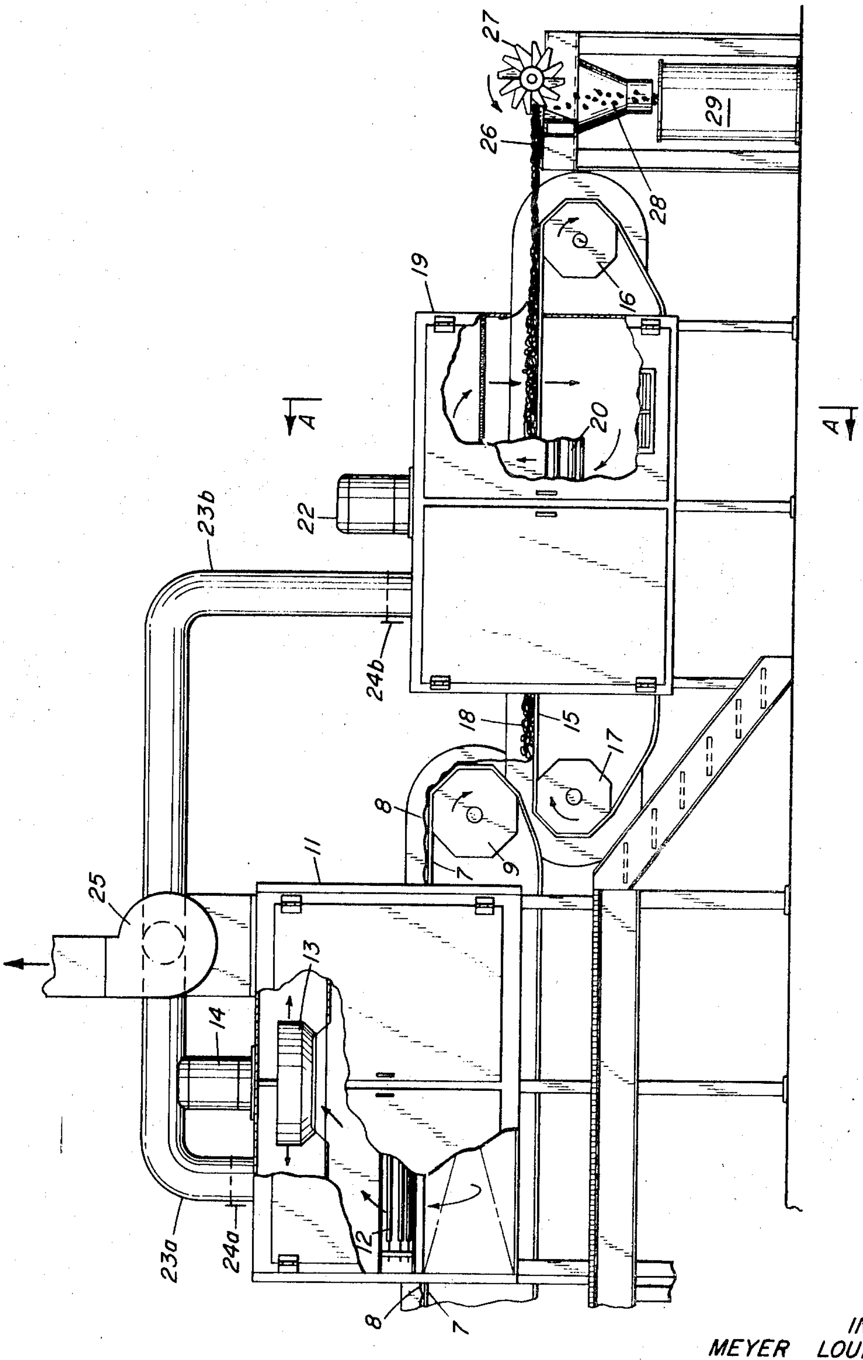

FIG. 1B

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PATENTED JAN 18 1972

3,634,944

SHEET 3 OF 4

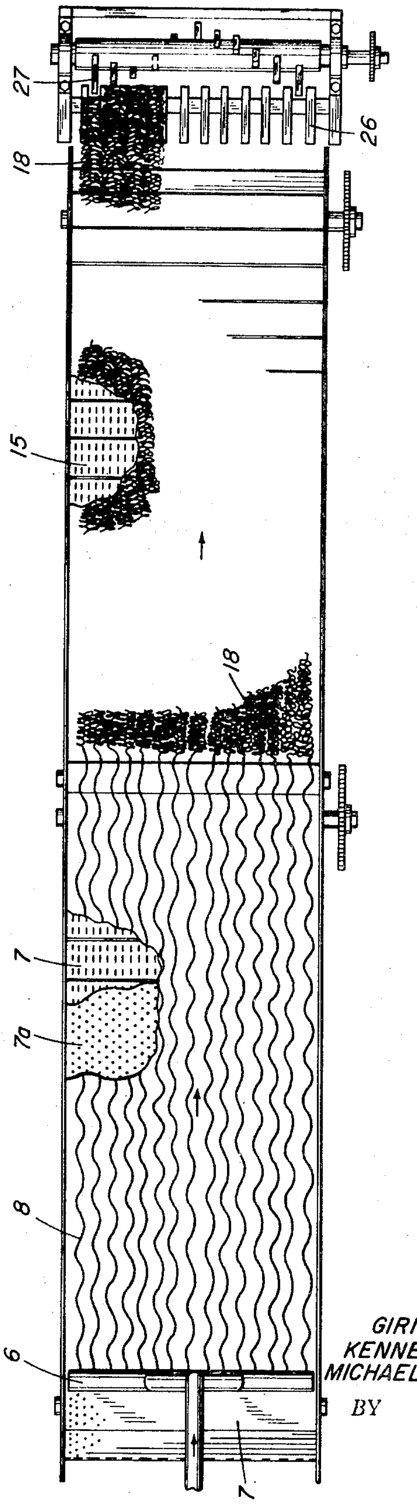

FIG. 2

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3,634,944

SHEET 4 OF 4

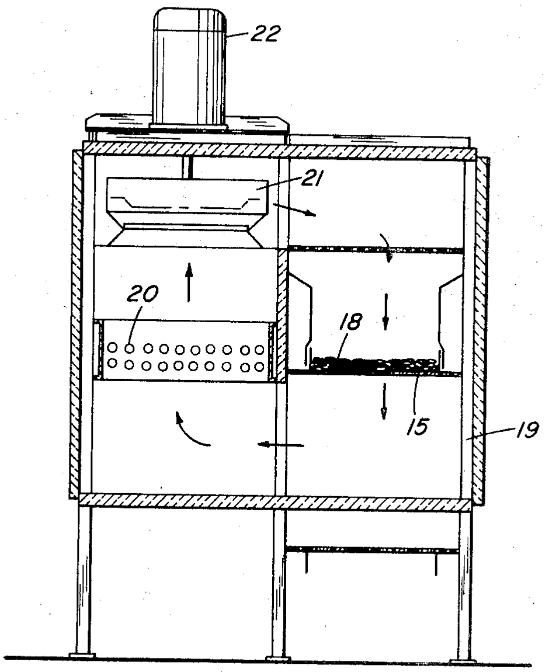

FIG. 3

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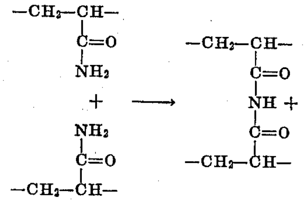
DRYING OF STICKY THERMOSENSITIVE HYDROUS GELS

The present invention relates to the continuous drying of sticky hydrous water-soluble substantially self-supporting acrylamide polymer gels to apparently dry nonadhesive and grindable state without rendering them water-insoluble, and to the grinding of said polymer to particulate free-flowing state likewise without rendering the polymer water-insoluble.

Water-soluble acrylamide polymers (vinyl polymers which contain at least 50 mol percent of acrylamide linkages) are currently produced on a major industrial scale and find use as wet and dry strengthening agents for paper and as flocculants in the purification of water. The polymers (hereinafter termed "polyacrylamides") are generally produced by polymerization in aqueous medium of acrylamide alone or in admixture with a molecularly minor amount of one or more water-soluble vinyl monomers copolymerizable therewith. The polymers are soluble in the aqueous medium. The polymers may be nonionic or they may be anionic or cationic (when they respectively contain anionic or cationic substituents). The immediate product of the polymerization is a clear hydrous gel so viscous as to be self-supporting i.e., so that a 1-inch cube thereof substantially maintains its shape when allowed to stand). The gel is very elastic and moreover it is intensely adhesive (about as much so as the glue of "Scotch" brand pressure-sensitive tape), and because of these properties it is very difficult to package in form acceptable to most consumers. Moreover, the gel is difficulty soluble in water and typically takes many days to dissolve. Experience has shown that the consumer prefers the polymer in apparently dry, particulate free-flowing (ground) form, one reason being that the polymer in this form dissolves comparatively rapidly in water. The gels are referred to possess low vapor and thermal permeability, and are very difficult to dry. When the gel is dried in normal manner in bulk at high temperatures, cross-linkages form on the surfaces of the gel which have been most strongly heated, causing portions of the polymer to become insoluble. One mechanism by which this cross-linking takes place is through formation of imide linkages as illustrated by the theoretical equation:



$$\begin{array}{c}\mathrm{-CH_2-CH-}\\ |\\ \mathrm{C{=}O}\\ |\\ \mathrm{NH_2}\\ +\\ \mathrm{NH_2}\\ |\\ \mathrm{C{=}O}\\ |\\ \mathrm{-CH_2-CH-}\end{array} \longrightarrow \begin{array}{c}\mathrm{-CH_2-CH-}\\ |\\ \mathrm{C{=}O}\\ |\\ \mathrm{NH}\\ |\\ \mathrm{C{=}O}\\ |\\ \mathrm{-CH_2-CH-}\end{array} + \mathrm{NH_3}$$

When the gel is dried at sufficiently low temperature to avoid formation of cross-linkages, an unduly long time and unduly large apparatus is required. Up to the present, for this reason, it has been considered impractical to dry the gel by the use of air (or other inert gas) alone.

The temperature at which the aforesaid cross-linkages start to form at a significant rate varies from instance to instance depending principally on the molecular weight of the polymer, the initial water content of the gel, the pH of the gel, the amount of monomer present, the molar proportion of the acrylamide units to any other units present in the polymer. The temperature at which these cross-linkages form in any instance to produce more than a negligible amount of water-insoluble matter (hereinafter termed "thermal degradation point") can be readily found by laboratory trial. For polyacrylamide itself the temperature is about 160° F. and for most acrylamide polymers the temperature is in the range of about 140°–195° F. From the point of view of efficient drying, these temperatures are low.

In the past, it has been proposed to comminute the gel and dry the gel to grindable state by the use of air of moderate temperature, (e.g., air having a temperature of 300° F.) in the hope that the water content of the gel would maintain the temperature of the gel sufficiently low to prevent formation of cross-linkages in insolubilizing proportions while providing an adequately fast rate of passage through the drier. It was found, however, that the temperature of the gel does It was found, however, that the temperature of the gel does not remain uniform during such drying but that the outer surfaces and particularly the corners of the comminuted gel reach the thermal degradation point sufficiently in advance of the point at which the gel as a whole becomes dry so that these surfaces become thermally degraded and insoluble before the gel as a whole becomes sufficiently dry to be ground. The insoluble portions of the polymer appear as gelatinous particles which may be sufficiently large to be termed "fish eyes" (resembling tapioca particles) and usually must be removed by filtration before the polymer solution can be regarded as meeting commercial standards.

The discoveries have not been made that the aforementioned gels possess slight but significant adhesiveness to polytetrafluoroethylene ("Teflon"); that these gels, when extruded in the form of cords of more or less circular cross section of critical area and contacted briefly with an inert drying gas having a temperature substantially above the degradation point of the polymer in the gel, rapidly form a skin which is flexible, uniform, and nonadherent; that polyacrylamide gel carrying the aforementioned skin possesses substantially no adhesiveness for polytetrafluoroethylene and can be readily stripped therefrom; that the resulting cords (having nonadhesive surfaces) can be automatically formed into thick gas-permeable mats which can be rapidly and safely dried by passage therethrough of an inert drying gas. By this means we have found that the sticky hydrous gels can be dried to hard grindable state in a compact drier at high throughput, that the resulting polymer is substantially completely (though slowly) water-soluble, and that this polymer becomes rapidly water-soluble when ground to powder form, as hereinafter more particularly described.

The nonadherent skin which is formed at the end of the first heating stage is temporary. The underlying body of the cords is highly hydrated, and when the cords are allowed to stand, water from the underlying body diffuses through the skin, rendering the surface substantially as tacky as it was before. In the process of the present invention, therefore, the second stage of drying is performed as a consecutive step, so that substantially no hydration of the skin occurs.

The present invention, therefore, is broadly a continuous process for the drying of a sticky, substantially self-supporting hydrous gel of a water-soluble thermosensitive polymer by extruding the gel as a plurality of parallel cords having a cross-sectional area between about 1 and 500 mm.^{2} upon a travelling foraminous belt having a release surface for the polymer, passing a nonreactive drying gas having a temperature from 100° F. below to 300° F. above the degradation temperature of the polymer in the gel only until the surface of the cords has become nonsticky, discharging the nonsticky cords from the belt, and contacting the cords with a nonreactive drying gas having a temperature sufficiently low that the aforementioned cross-linkages do not form.

In the process, the cross section area of the cords is critical. If the area is much larger than 500 mm.^{2}, the second stage of the drying will take too long, the apparatus will not be compact, and the skin of the cords may become insoluble before the cords have been dried to grindable state. If the area is much less than 1 mm.^{2}, the power requirements of the apparatus increase sharply because it is difficult to extrude a tough rubbery gel in the form of small diameter threads, and since the volume/surface area ratio will be small, there is danger that during the drying, the temperature of the skin may be carried above the thermal degradation point. As the result of a large number of trials we have found it practical in the first stage to extrude the gel as cords having a cross section area in the range stated and to convert the surface of the cords into a nontacky skin by contact with a hot current of air, and then to form the cords into a thick mat and dry the mat with air having a temperature not substantially in excess of the thermal degradation point. Being nontacky and substantially self-supporting, the mat which is formed has an open structure permitting the passage of the drying air to every part.

The invention is described more in detail in the drawings, wherein

FIG. **1A** is an elevation showing schematically the reactors in which acrylamide polymer gel is formed and the end of the first travelling belt on which the gel from the reactors is discharged;

FIG. **1B** is an elevation partially broken away showing schematically the parts of the first and second belts on which the gel is dried, and the comminution of the dried polymer to coarse particulate state;

FIG. **2** is a plan view, partly in section, of the two belts and comminution section of the apparatus shown in FIGS. **1A** and **1B**; and

FIG. **3** shows partially in elevation and partially in section the apparatus for supply of hot inert gas to the second drying belt along line A—A' of FIGS. **1B** and **2**.

In the figures the same reference numerals and letters designate the same components.

In FIG. **1A** the acrylamide polymer hydrous gel is formed in polymerization chambers **1***a* and **1***b* working in parallel, and is supplied through valves **2***a* and **2***b* working alternately through pipes **3** and **4** to screw pump **5** provided with pipe **5***a* at its exit end for supply of special additives. The gel is supplied under pressure to header **6** extending across the width of travelling jointed steel belt **7** carrying release surface **7***a* where it is extruded in the form of cords **8** having a cross section area of 1 mm.^{2} to 500 mm.^{2} and preferably in the range of 25–50 mm.^{2}.

As is particularly shown in FIG. **1B**, **2** and **3**, belt **7** is carried forward by drive roll **9** and idler roll **10** and carries the cords into cabinet **11** where they are contacted with hot inert gas supplied by passage of the gas through conventional heating coils **12** and distributed by fan **13** driven by motor in housing **14**. The gel cords now carrying a nontacky skin are discharged by gravity from nonadherent belt **7** and fall upon travelling belt **15** carried forward by drive roll **16** and supported by idler roll **17**. The speed of belt **15** is one-tenth that of belt **7**. As a result the cords form a mat **18** about 10 times the thickness of individual cords **8**.

Belt **15** carries the mat into low-temperature drying cabinet **19** where the mat is dried by passage therethrough of inert drying gas heated by coils **20** and circulated by fan **21** driven by motor in housing **22** in the same manner as the gas in cabinet **11**.

The humidity and solvent content of the gas is controlled by bleeding of gas through ducts **23***a* and **23***b* provided with dampers **24***a* and **24***b* leading into exhaust fan **25**.

The mat of acrylamide polymer gel cords, now in hard, rigid fracturable state, is continuously discharged from belt **15** upon cutting table **26** where rotating chopper blades **27** fracture it into pieces **28** of grindable size, which are collected in bin **29**.

The pieces are then ground to a particle size range (e.g., 90 percent through 40 mesh) which permits the polymer to dissolve rapidly when stirred with water. Any convenient grinder (for example, a hammer mill) may be used. In continuous operation the pieces entering the grinder are close to their thermal decomposition point, and the grinding operation further elevates the temperature of the polymer. The grinding temperature is, therefore, kept below the thermal decomposition point by the introduction of cold gas, and we have found it most convenient to supply such gas by spraying liquid nitrogen or carbon dioxide into the grinding chamber.

The numerical constants of the apparatus vary from instance to instance and do not conform to a simple rule. In each of the stages the principal variants are the speed of the belt; the diameter, water content and cross section configuration of the cords on entrance; the temperature, velocity and relative humidity of the air in the two chambers; and the supply (or nonsupply) of radiant (e.g., infrared) energy to the cords to provide a penetrating heat. The constants cannot be easily predetermined but can be determined by trial, employing the data of the examples as the start. However, we have found that in the second stage the temperature of the heating gas can be as much as 50° F. above the thermal degradation point without formation of an objectionably large proportion of insoluble matter in the polymer. In general, the larger the cross-sectional area of the polymer the lower should be the temperature of the second stage drying air. The maximum temperature can be used in the case of cords having cross-sectional areas in the bottom of the range (e.g., less than about 50 mm.^{2}). At the top of the range (e.g., in the range 100 mm.^{2} to 500 mm.^{2}) the temperature of the drying gas should not be more than a few degrees above the thermal degradation point.

In the process, the dwell time of the polymer in the second drying stage need be no longer than is required to render the polymer conveniently grindable, i.e., with a water content of 5 to 15 percent. Insolubles tend to form in the polymer when the dwell time is extended, and there is no offsetting advantage.

The process of the present invention is usefully employed with gels of water-soluble acrylamide polymers which contain a material selected from the group consisting of cyanamide, guanidine, hydrazine, mercaptosuccinic acid, succinimide, thioglycollic acid, urea, and mixtures of these materials as agents which inhibit the formation of cross-linkages in the polymer. Without these materials, the dried acrylamide polymer product of the present invention generally contains a trace of water-insoluble material. With the above-mentioned materials present in effective amount (usually 0.02–2 percent on a dry basis), the polymer product is generally completely soluble in water. If desired, the materials may be introduced by uniformly mixing them in dry powdered state into the gel and allowing the gel to stand. The particles dissolve and become uniformly distributed through the gel. Alternatively, the materials may be incorporated into the gel by dispersing the material in solution state throughout the gel and allowing a few minutes for the liquid to diffuse through the gel. Alternatively still, the materials may be introduced into the solution of monomeric material from which the polymer is formed. Details are provided in copending application Ser. No. 878,883, filed on Nov. 21, 1967 by M.N.D. O'Connor. The presence of these compounds in the gel during the drying step confer the added benefit of converting any monomeric acrylamide present to other and nontoxic form, as is disclosed in said application. Monomeric acrylamide is toxic, and to this extent the materials recited above have the property of acting as detoxifying agents.

The grinding step is facilitated by the presence of a uniformly distributed amount of a water-soluble salt in the polymer. The salt increases the throughput of the grinder without increasing power requirements on the one hand, and permits the output of the grinder to be increased with less power than would be expected. Suitable salts for the purpose include sodium sulfate, potassium sulfate, sodium acetate, disodium phosphate, sodium nitrate, aluminum sulfate and magnesium sulfate.

The invention is further described by the examples which follow. These examples are preferred embodiments of the invention and are not to be construed in limitation thereof.

EXAMPLE 1

The following illustrates a typical embodiment of the present invention performed in apparatus similar to that described in the drawings.

An aqueous hydrous gel of polyacrylamide having a molecular weight of about 10 million, a water content of about 90 percent by weight, a thermal degradation temperature of about 160° F., is delivered under a pressure of about 100 lb./in.^{2} by a screw pump to an extruder similar to that shown in FIG. 2. The extruder produces a parallel array, about 10 feet wide, of cords one-fourth inch in diameter and one-half inch apart. The extruded cords are about as sticky as surgical adhesive tape, they are very flexible, and resemble elastic band rubber. The cords fall without losing shape upon a horizontal stainless steel belt (10 feet wide and 2 inches below the extruder) carrying a polytetrafluoroethylene fabric surface and moving 10 feet per minute. This speed is slightly faster than the speed at which the cords leave the extruder. The cords adhere slightly to the polytetrafluoroethylene surface, so that the cords are slightly stretched and lie flat on the belt in parallel array. The cords are carried on the belt into a drying chamber 50 feet long where they are contacted with air at 330° F. flowing through the chamber at a speed of 300 feet per minute. On emerging from the chamber (dwell time 5.0 minutes) the cords have a water content of about 70 percent by weight. They carry a nonadhesive skin but are still flexible, elastic, clear and glossy, having soft and sticky centers.

The cords are then discharged upon a horizontal stainless steel belt moving 5.0 inches per minute and are carried in the form of a thick open-structured mat about 4 inches thick into a drier chamber 50 feet long where they are contacted with dry air at 160° F. On emerging from the chamber (residence time about 2 hours) the cords have a water content of 5–10 percent by weight and a diameter of about one-eighth inch. They are opaque, stiff, and uniformly hard and dry.

The mat is then passed under a rotary chopper where it is broken into pieces roughly one-half inch in largest dimension, which are fed into a hammer mill, the interior of which is maintained at 120° F. (well below the thermal degradation temperature of the polymer) by liquid nitrogen sprayed into the interior through pipes passing through the sides of the grinding chamber.

The product from the hammer mill has a particle size of 100 percent —18 mesh, 56 percent —30 mesh and 18 percent —100 mesh. It dissolves completely in 30 minutes in water at one-half percent concentration at 70° F. No "fish eyes" are visible in the solution. Filtration of the solution shows that the product contains a trace (less than 0.5 percent) of insoluble matter.

EXAMPLE 2

The following illustrates the production of a similar dried polyacrylamide powder in more compact apparatus resulting from the use of a gas for the second drying step which has a temperature above the thermal degradation point of the polymer.

The procedure of example 1 is repeated using the same gel except that the length of the heated zone in the second drying oven is decreased to 35 feet, and the temperature of the second drying air is increased to 190° F. The product is substantially the same as the product of example 1. Evidently, in the second drying stage evaporation of water from the gel kept the temperature of the gel below the thermal degradation point of the polymer.

EXAMPLE 3

The following illustrates a method for the production of a dry acrylamide polymer which contains no detectable amount of insoluble polymer or other insoluble polymer.

The procedure of example 1 is repeated using the same gel except that the gel has a uniform dissolved content of 1 percent of urea based on the weight of polymer therein, as disclosed in said copending application.

The ground product dissolves completely in water at a concentration of one-half percent and a temperature of 70° F. and the solution contains no insoluble matter.

EXAMPLE 4

The following illustrates a process according to the present invention in which a polyacrylamide gel is dried and ground to a form in which it is rapidly and completely water-soluble and in which some of the amide substituents are converted to anionic (carboxylic) form.

The procedure of example 1 is repeated using the same gel except that 0.07 mol of sodium hydroxide (as a 15 percent by weight aqueous solution) per acrylamide linkage in the polymer is metered into the gel as it leaves the screw pump. About 7 percent of the amide substituents of the polyacrylamide hydrolyze during the subsequent mixing and drying steps, so that the product corresponds to a 93:7 molar ratio anionic acrylamide:acrylic acid copolymer. The product, after grinding, dissolves rapidly in water at one-half percent concentration and 70° F. to form a solution which contains less than 0.5 percent insoluble matter.

EXAMPLE 5

The following illustrates the process of the present invention on a gel wherein the polymer contains cationic substituents.

The procedure of example 1 is repeated except that the polymer in the gel which is dried is a 95:5 molar ratio acrylamide:2-(dimethylamino)ethyl methacrylate copolymer having a molecular weight of roughly 10,000,000. The product, after grinding, contains less than 0.5 percent insolubles.

EXAMPLE 6

The following illustrates the process of the present invention wherein the gel when dried contains a water-soluble salt as agent improving the grindability of the gel.

The procedure of example 1 is repeated except that sufficient of a 25 percent solution of sodium sulfate is metered into the gel as it leaves the screw pump to provide 15 percent of Na_2SO_4 based on the dry weight of the polymer. The dry polymer is substantially the same, but is more readily ground.

EXAMPLE 7

The following illustrates the process of the present invention applied to the drying of a preformed anionic acrylamide polymer gel with introduction of supplementary polymerization catalyst to detoxify any monomer present. The gel is similar to that of example 1, but the polymer is a 90:10 acrylamide:acrylic acid copolymer having a molecular weight of about 5,000,000.

The procedure of example 1 is repeated, except that into the gel transport line between the transfer pump and the extruder is pumped a solution of alkali metal persulfate ($K_2S_2O_8$) sufficient to supply 50–80 p.p.m. of the persulfate based on the dry weight of the polymer except that in the first stage of drying the cords are contacted with air at 265° F. for 9 minutes and in the second stage the cords (in matted state) are contacted with air at 180° F. for about 105 minutes. The dry polymer contains 0.01 percent by weight of water-insoluble matter.

EXAMPLE 8

The following illustrates the procedure of the present invention wherein drying and a large amount of hydrolysis occur during the drying.

The procedure of example 1 is repeated except that sufficient concentrated sodium carbonate solution is metered into the gel on exiting from the screw pump to provide 35 percent hydrolysis of the polyacrylamide, and in the first stage the gel is contacted with air at 265° F. for 21 minutes and in the second stage with air at 175° F. for 106 minutes. The product contains 9.8 percent water by weight, is readily grindable, and contains no detectable amount of insolubles.

We claim:

1. Continuous process for drying a sticky water-soluble, substantially self-supporting hydrous gel of a water-soluble acrylamide polymer without rendering said polymer water-insoluble, which comprises extruding said gel as a plurality of cords having a cross-sectional area between about 1 mm.^{2} and 500 mm.^{2} upon a travelling belt having a release surface for said polymer; contacting said cords on said belt with a non-

reactive drying gas having a temperature from 100° F. below to 300° F. above the degradation point of the polymer in said gel only until a nonsticky skin has formed on said cords; discharging said cords from said belt; and contacting said discharge cords with a nonreactive drying gas having a temperature not more than 50° F. above the thermal degradation point of the polymer in said gel until said cords are sufficiently dry to be grindable.

2. A process according to claim **1** wherein the cross section area of the cords is in the range of 25 mm.^{2} to 50 mm.^{2}.

3. A process according to claim **1** wherein the temperature of said first gas is 50°–100° F. above the degradation point of the polymer in said cords.

4. A process according to claim **1** wherein the polymer contains 0.02–2 percent by weight (dry basis) of a material selected from the group consisting of cyanamide, guanidine, hydrazine, mercaptosuccinic acid, succinimide, thioglycollic acid, urea, and mixtures thereof, as agent inhibiting formation of cross-linkages in said polymer.

5. A process according to claim **3** wherein the cords are discharged from said first belt upon a second belt having a speed about one-fifth to one one-hundredth that of the speed of said first belt.

6. A process according to claim **1** wherein the temperature of said second drying gas is at about the thermal degradation point of said polymer.

7. A continuous process for converting an aqueous, sticky, substantially self-supporting hydrous gel of a water-soluble acrylamide polymer to dry, particulate and readily water-soluble state, which comprises drying said gel to grindable state by the method of claim **1**, and grinding said gel in an inert atmosphere having a temperature sufficiently low to maintain the temperature of said polymer during said grinding below its thermal degradation point.

8. A process according to claim **7** wherein the temperature of said atmosphere is maintained sufficiently low by evaporation of liquid nitrogen.

9. A process according to claim **1** wherein the gel contains a water-soluble alkaline material as hydrolyzing agent for amide substituents therein.

10. A process according to claim **9** wherein the alkaline material is sodium hydroxide.

11. A process according to claim **7** wherein the gel contains a water-soluble inorganic salt as agent improving the grindability of said gel.

12. A process according to claim **11** wherein the salt is sodium sulfate.

* * * * *

1. The following is an examiner's statement of reasons for allowance: The patent of Bronstert(EP 101,893) appears to teach a tapered reactor but not solution polymerization of AA, or MAA or their esters.

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

T. Weber
September 28, 1996

THOMAS R. WEBER
PRIMARY EXAMINER
GROUP 1500

pieces that can remain stuck in the reactor. While this may sound trivial in hindsight, the process involves pushing viscoelastic rubber-like polymer gel through a continuously constricting taper, and we know that squeezing through a taper offers more resistance than pushing down a non-tapered tube. Until the inventors built a reactor with a conical taper and used inert gas to remove the rubber-like polymer gel, there was no way to know if the claimed process would work. And even Dr. Freeman acknowledges that "[t] here are a number of challenges associated with synthesizing polymers that impact the choice and design of polymerization reactors." Freeman Decl. ¶ 27. A person of ordinary skill would not have had a reasonable expectation of success in achieving removal, or for that matter any reasonable amount of discharge, of a rubber-like polymer gel based on the combination of the '597 patent and the '944 patent.

148. The discharge problems with viscoelastic rubber-like gels are squarely addressed using pistons (GB'028 and JP'181) or a separating liquid (EP '709) in a cylindrical reactor. Also, JP '181 expressly discouraged the use of inert gas due to the bypass problem. In contrast, the '944 and '597 patents provide no information to suggest a conical taper and inert gas can somehow promote discharge of rubber-like polymer gels, much less effect their removal. Accordingly, a person of ordinary skill could not have predicted that removal of the gel could be achieved.

unsatisfactory because reactors using pistons were particularly susceptible to "the high wear which is caused during operation by friction between the inner wall of the reactor and the piston." '329, 1:25-32. The issues of high wear and friction in the reactors caused by pistons led to increased maintenance required for the reactors and reduction of the useful life of the reactors.

202. The prior art reflects that those who attempted to use inert gas to remove polymer gel from a reactor found that it was not a viable option. The JP '181 reference stated that inert gas discharge "has problems" including "difficulties in discharging the product as the inert gas short path through gaps between the inner walls and product." JP '181 ¶ 3. The inventors in EP '709 found that without some liquid medium between the polymer gel and the walls of the reactor, it was difficult to achieve a complete and efficient discharge of a polymer gel product with gas pressure. EP '709 3:22-29; 6:22-27.

203. Furthermore, a person of ordinary skill would have had a wide variety of alternatives to using a piston. For example using a conveyer belt as a reactor (*see* Ex. 2011, U.S. 4,138,539), using a tubular reactor with static mixers (*see* Ex. 2012, U.S. 4,110,521), using a wiped-surface reactor that contains rotor with a wiper to wipe the inside surface of the reactor (*see* Ex. 2013, U.S. 4,619,979), using a specially configured mixture that prevents polymer from building on the

Q. And then you referred to that in your 14:13:26
declaration, did you not? 14:13:30
A. Yes. 14:13:31
Q. And then it refers to JP '181, which you 14:13:31
also referred to in your declaration. True? 14:13:36
A. Yes. 14:13:40
Q. And then it refers to EP '709, which 14:13:40
we've been talking about, and of course you referred 14:13:44
to that in your declaration, right? 14:13:46
A. Yes. 14:13:48
Q. And then it refers to the fourth of 14:13:48
these prior art patent documents as U.S. Patent 14:13:50
No. 5,081,215, which you never referred to in your 14:13:54
declaration. Why is that, sir? 14:13:58
A. Because I was unaware of it until -- 14:14:16
until, I believe, it was brought up at our -- 14:14:25
Dr. Carson's deposition, which was after my depo- -- 14:14:32
declaration was written. 14:14:40
Q. I don't quite understand what you mean 14:14:41
by "unaware of it." You did read the '329 patent, 14:14:46
did you not? 14:14:49
A. Yes. 14:14:49

Q. And it discusses and describes the '215 14:14:49
patent. 14:14:54
A. Yes. 14:14:55
Q. So you were aware of it? 14:14:56
A. But I had not read it. 14:14:56
Q. Why not? It's a BASF patent document, 14:14:58
isn't it? 14:15:00
A. Uh-huh. 14:15:01
Q. So whatever is said in there should go 14:15:02
just as well for what they say in the '329 patent. 14:15:05
It's the same people, right? 14:15:08
MR. RIDDLE: Objection to this line 14:15:09
of questioning for the same reason that this is 14:15:11
beyond the scope of the petition or the grounds 14:15:14
instituted by the Board. It's irrelevant and 14:15:17
beyond the scope of proper cross-examination. 14:15:21
MR. BRADY: I believe it goes to 14:15:25
motivation, reasonable expectation of success, 14:15:26
reasons to combine, what a person of ordinary 14:15:33
skill in the art knew and would do, and it's 14:15:37
admitted prior art discussed by the same patent 14:15:39
owner in the '329. 14:15:41

showing inventor Shigeo Inoue and assignee Sanyo Chemical Industries). JP ’181 indicates that the gas discharge approach mentioned in passing by the ’597 patent was prone to problems and suggests such approaches actually were unsuccessful. Appx2602-2603 ¶ 60, Appx2628-2629 ¶ 138.

The ’215 patent. The ’215 patent describes a process for making a type of plastic known chemically as polyarylene etherketones. Appx1897, 1:5-7. The process is a two-step, two-reactor process where gas pressure is used to move material between and within the reactors to a discharge aid, located within the second reactor. Appx1897, 1:63-2:2, Appx1898, 4:31-38. The discharge aid is described as a piston or screw. Appx1898, 3:55-63, 4:3-9; Appx336-337. While the ’215 patent is prior art under 35 U.S.C. § 102(e), it also is a BASF invention and therefore not citable for purposes of an obviousness inquiry according to 35 U.S.C. § 103(c). For its part, the ’329 patent provides a brief description of the’215 patent:

> U.S. Pat. No. 5,081,215 discloses a process for preparing polyether ketones in which the polycondensation is carried out by a two-stage process, the polycondensation being completed in the second stage and the plastic polymer matrix containing included aluminum chloride particles obtainable being discharged from the tubular reactor virtually without residue by injecting an inert gas.

Appx27, 1:65-2:4.[3]

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[3] Since the ’215 patent was mentioned in in the ’329 patent, the Board discussed it in its final decision. In its Reply, SNF raised twelve other new references, offered a new 55-page expert declaration, and multiple new arguments. All of this new

United states Patent and Trademark Office

SNF Holding Company

v.

BASF Corporation

Transcription of Audio

Federal Circuit Argument

Chief Judge Prost: Hope Houston is doing better. Wasn't that the reason for a delay? Ok? The case for argument this morning is 16-2565 SNF Holding versus BASF. Mr. Dabney.

Mr. Dabney: I reserve 5 minutes. Chief Judge Prost and may it please the Court. The Board in this case committed 2 errors that call for reversal. First, in its final written decision, the Board for the first time announced a construction of the claimed invention that differs substantially from what the patent, the prosecution history and the Board's own institution decision had identified as the claimed invention.

Judge Wallach: The '215 patent is directed to a different of type of polymerization reaction than '329, am I correct?

Mr. Dabney: It is, you honor. However, the—at the time of prosecution, that was exactly the point that the examiner made to distinguish the '215 patent from the claimed subject matter—exactly that, that the gel in the '215 reactor vessel was an anhydrous gel as opposed to a hydrous gel. So the reason for allowance stated in the patent on page 701 of the joint appendix is "the prior art shows a tapered reactor but not solution polymerization of AA, acrylic acid, MAA methacrylic acid or their esters. So—

Judge Wallach: So is '215 then not relevant prior art?

Mr. Dabney: No, '215 is crucially relevant prior art because it shows what the state of the art was, number 1, and more importantly, it contained a crucial admission on the part of the patent owner as to what was known in the art at the time. I think the best way to understand what's going on this case is to turn to pages 17 and 18 of the Board's decision which is in the joint appendix and with the Court's permission, I will—I have drawn a sketch of the figure that's on page 18 of the Board's decision, which I think will aid the Court in following along with the argument. What the patent says in columns 1 and 2 is that before the claimed invention was made, it was known you had to use a tubular reactor having a conical taper, having an inlet for filling it with reactants, having a diameter ratio of between 2 to 1 and 10 to 1, having an inlet for gas injection and in a mode of operation, quote, "the plastic polymer matrix being discharged from the tubular reactor virtually without residue by injecting an inert gas."

Chief Judge Prost: Well I think turning to those—As I understood what the Board was saying was that the reason this is not helpful to you or as helpful as you might like, is that they've got that number 6 there which is the discharge aid. So that this is another factor and which is not present in the claims and that's how the Board differentiated the '215, so what's wrong with that—I mean—

Mr. Dabney: Your Honors—

Chief Judge Prost: It says bottom of 18—

Mr. Dabney: Your Honors—

Chief Judge Prost: "Both inert gas pressure and the discharge aid located," and the discharge aid is what they relied on as differentiating the two.

Mr. Dabney: There's 2 crucial points about that. First of all, the '215 patent was a reference that the patent owner's expert never mentioned in his expert report. At the time of the patent owner's—

Judge Wallach: Well show me a reference that expressly promotes a conical taper as being advantageous for any purpose relevant to the types of polymers claimed in '329.

Mr. Dabney: On page 501 of the joint appendix, column 3, line 70, there is a specific disclosure of a tubular reactor being used to make the very type of polymer—

Chief Judge Prost: Hold on. You are going too fast. 501, column 3—

Mr. Dabney: 501—page 501—

Chief Judge Prost: Column 3—

Mr. Dabney: Column 3, line 70—

Chief Judge Prost: The first example—

Mr. Dabney: There is a statement starting at line 70 "The resulting gel-like polymer solution was discharged from the vessel through the outlet by the use of nitrogen gas pressure, and the description of the dimensions of that make it clear that the reactor vessel is cyclindroconical. So, to the extent that it was ever a requirement, which was never suggested in the patent, never suggested in the prosecution history, never suggested in the Board's institution's decision, that the innovation here was not the substitution of a hydrous gel for an anhydrous gel in the reactor but rather was injecting gas pressure for a length of time sufficient to bring the reactor vessel to a state of substantial emptiness.

Chief Judge Prost: Ok. Either I haven't asked the question right or you're not answering it because the—it seems to me the basis upon which the Board relied for differentiating the '215 is this number 6 thing, that it wasn't just—it used both the inert gas pressure and a discharge aid and then on 18, 19 they go on to say, indeed the discharge aid tends to support the patent owner's argument that what—that it wasn't the pressure alone that did it. So that's the basis upon which the Board differentiated the '215. So what is wrong with that?

Mr. Dabney: It's because that wasn't the patent owner's argument. The patent owner's argument is summed up on page 59, paper 20 of exhibit 2016 in the record.

Chief Judge Prost: You are going to have to give me a better—What—

Mr. Dabney: Paper 20. It is in the record, it is not in the joint appendix but what the patent owner said, quote, "Prior to the invention of the '329 patent, conical tapers and inert gas had never been used to remove polymer gels from reactors." And the other side's expert in exhibit 2016, in the record, in paragraph 202, said "The prior art reflects that those who attempted to use inert gas to remove polymer gel from a reactor found it was not a viable option." When they responded to the petition they took the position that this reference was not citable in an obviousness combination and on page 10 of their brief, they make a crucial error. On page 10 of their brief to this Court, they say, quote, "While the '215 patent is prior art under 35 U.S.C. 102(e), it is also a BASF invention and therefore not citable for purposes of an obviousness inquiry according to 35 U.S.C. 103(c). Wrong! This patent issued in 1997. At that time, 103(c) did not cover 102(e)–103 references. So what happened here is that all throughout the Board proceeding, the patent owner took the position that this reference was not rightly considered by the Board. Their expert didn't put in any evidence. What they did is, they tried to impeach what their own patent said about the state of the art, what was known in the art, with general oral testimony by an expert who said "This had never been done," that said, "use of conical tapers and inert gas had never been used to remove polymer gels." So the patent owner made this sweeping argument which was based on error that this was not rightly considered, an error repeated in their brief to this Court and so when we came back in and said "How can you possibly make this statement that conical tapers and inert gas had never been used to remove polymer gel from a reactor?" the answer was "Their expert hadn't considered this." So—

Judge Taranto: Can I ask, if this was prior art why wasn't it one of the principle prior art references on which you based your challenge?

Mr. Dabney: Because the patent gave no inkling that what made this nonobvious was the idea of injecting gas pressure for long enough that the reactor achieves a state of substantial emptiness. What the patent identified as the innovation, what the examiner said was the innovation, was the monomer. This was old. They had used this exact process to make anhydrous gel intermediate. They said our innovation here is not making just any high molecular weight polymer. This claims—every claim specifically recites making high molecular weight polymer using water soluble, monoethylenically unsaturated monomers in aqueous solution.

Judge Wallach: Was 103(c) cited below?

Mr. Dabney: I don't know that it was cited below. It was baked in to the patent owners' presentation. They submitted expert—I invite the Court to read exhibit 2016. That is the the sole evidence.

Judge Wallach: Well look—No, no. I asked you a question.

Mr. Dabney: To my knowledge, it was not cited below.

Judge Wallach: So you didn't discuss it below, that it didn't exist at the time?

Mr. Dabney: We—until the final written decision, this—I mean the Board in its institution decision never bought the argument that this wasn't rightly considered. And then during the proceeding, the patent owner simply took the position that this wasn't rightly considered and said its expert didn't consider it.

Judge Wallach: Let's talk about the claim construction of "removing"— we don't have a ton of time, you know.

Mr. Dabney: Yes. Our position on that is very simple—

Judge Wallach: No, no. I have a question for you. Thank you. Ok. On page 31 of the blue brief, you make your interpretation argument, and you say that "removing" has its ordinary meaning as "discharging," and you say specifically "removing describes an active process of removal, not one that has been completed. It synonymous with discharging." Is that any amount no matter how tiny?

Mr. Dabney: "Removing" as an operation begins when it begins, so the patent says "ejecting, removing, discharging, pushing out." It's describing what is happening when the gas pressure is applied. So for instance, in a continuous process, the reactor, optimally, is being filled—

Judge Wallach: Is one percent enough to qualify as removal?

Mr. Dabney: If you perform that element for only 5 seconds, yes, because "removing" is describing an operation. What is the machine doing at this point? It's polymerizing. Ok. What is the machine doing at this point? It is in the process of removing what's in the reactor, so if you think of removing the way the patent describes it, as how are you getting it out? It's not with the piston. It's not with an auger. It's being pushed out by injection of inert gas so if you leave the inert gas on for five seconds, not that much is going to come out but if you leave it on for 3 hours, a lot of it will come out. But this idea that patentability depends on how long you put the gas pressure on, so whether its—at some point you reach a state of substantial emptiness, that was never—prior to 2015, there was never any suggestion that any patentability depended on what length of time you put the gas pressure on.

Chief Judge Prost: No no no, but what the Board, I think relied on, is that the patent is chocked full of examples, and you're right those examples don't—it's confusing because the examples use the word "discharge" and not "removal," but what they say about "discharge" is "discharging without residue."

Mr. Dabney: Yes, which is exactly what was in the prior art. The fact that gel doesn't stick to Teflon, was not a new discovery in 1995. The patent '215 says if you want to get the gel out, you line it with Teflon, and optionally you can include a displacement aid here so that the gas doesn't tunnel down through the center, but one of the glaring errors that the Board made in analyzing this patent—because the patent owner didn't put any evidence in, and the board didn't accept the erroneous argument that it is not properly considered—is that they ignored the evidence of petitioner's expert appearing in 1878 to 1882 that this shows the exact mode of operation as is claimed in the '329, and the Board made—conflated and confused the displacement device, this item 5, with the discharge aid, which is an apparatus that is downstream of the reactor. They said the discharge aid was within the conical portion of the reactor. There is no testimony—

Chief Judge Prost: Whoa, whoa whoa, what are we talking about, where are we in the Board's analysis?

Mr. Dabney: On pages 18 and—on page 18 and 19 of the Board's decision. It, it, it—at the last 2 lines on page 18 it says, "Given that the '215 discloses the use of both inert gas pressure and a discharge aid located within the conical portion," well the thing that's located within the conical portion is the displacer 5. Displacer 5. The discharge aid is down here—

Chief Judge Prost: Ok

Mr. Dabney: And by the—so that's just the glaring factual error which a Board can fall into when they're doing it all on their own, they're not relying on evidence submitted by the parties, they are doing this all on their own, and by the way, the Board said we're not going to credit this because the patent owner's combination doesn't include a discharge aid. Well that is another factual error. Had we known that they were going to do that, which we didn't know in advance, we would have pointed out that the '597 patent, the second reference, is all about a downstream apparatus that is the same as the discharge aid. It is—

Chief Judge Prost: Ok what are you talking about now?

Mr. Dabney: If we—if you turn to page 501 again, 501, which is the page of the patent, if you look, actually if you turn to 499—

Judge Wallach: Of the '597?

Mr. Dabney: '597. Yes. If you look at page 499, 499 is a picture of an auger that is receiving the feed of gel from the polymerization reactor. And the whole point of this patent is to provide an apparatus that is the same as what the '215 patent refers to as a "discharge aid." In this particular case, it is a horizontally-mounted auger that applies negative pressure to what's coming in and pushes it out through what's something like a meat grinder to chop it up. It's the same function, so—

Judge Taranto: What was the specific column line material that you read from page 501?

Mr. Dabney: Line 70, line 70. On column 3, column 3 on page 501 says "the resulting gel-like polymer"—

Judge Taranto: I'm sorry, you're speaking too fast for me.

Mr. Dabney: I'm sorry.

Judge Taranto: '597 patent.

Mr. Dabney: Yes.

Judge Taranto: Page 501 of the joint appendix.

Mr. Dabney: Yes.

Judge Taranto: Column 3, line what?

Mr. Dabney: 70. 70. "The gel-like polymer." "the," not "some." "The" gel-like polymer. And the last point I'd like to make before sitting down—

Chief Judge Prost: Wait, wait. Can we just work that story a little more. That sentence, the resulting—what are you trying to say?

Mr. Dabney: The resulting gel-like polymer—the suggestion is made, and again we had no notice, we had no APA notice that this was the claimed invention, that this was anything we—

Judge Wallach: If half the polymer was discharged would that be "the"?

Mr. Dabney: Say that again?

Judge Wallach: I said if half of it was discharged would that be "the"?

Mr. Dabney: I would say not.

Judge Wallach: Really?

Mr. Dabney: No. Well, I mean "the" is a definite adjective. It is talking about a specific quantity, I would suggest.

Judge Wallach: All?

Mr. Dabney: "The" means "all," I would say in this context, yes. That's what our expert said. The last point I'd like to make if I could is that the patents in this art, the '215 patent, the '329 patent, and this patent up at the top of that same column on page 501, you'll notice it says "also tubular-shaped reactors are quite suitable for this purpose." The word "tubular" in this art is routinely used to describe tubular reactors that have varying diameters so don't be misled into thinking that "tubular" in this art means a cylinder only. These reactors are all characterized as tubular, including in the '329 patent itself.

Chief Judge Prost: We're into rebuttal. Why don't you sit down and we'll hear from BASF.

Mr. Dabney: Thank you your Honor.

Mr. Riddle: May it please the Court. Because the analysis set forth in *Philips* strongly favored the "substantially all" construction, the PTAB reached the correct claim construction for the term "removing" in the '329 patent. And on the issue of claim construction—

Chief Judge Prost: Is "removing"—is the way you're defining "removing" based on the example "discharging without residue"? Is that the same thing?

Mr. Riddle: It's not exactly the same thing, I believe your Honor. I think for removing, if you look to the teachings of the specification, what does the '329 patent teach us? When it uses the word "discharge," there's always a qualifier with it. And the whole purpose—

Chief Judge Prost: And the qualifier is—

Mr. Riddle: —is "complete" discharge or "substantially complete" in the terms of the examples.

Chief Judge Prost: I mean, why—I think my question was, doesn't it use the words "discharge without residue" and is that the same as "remove"?

Mr. Riddle: Yes, and—

Chief Judge Prost: Yes, and I'm looking at the examples and you're talking about what it says about "discharge," and the words used are "discharge without residue." Are you disagreeing with me?

Mr. Riddle: No, in some cases, it certainly does say "discharge without residue" and in some cases in the examples—example 14 in particular says "discharge with insignificant residue." And at the very beginning of the patent, the way the problem set forth that this invention was aimed to solve was the problem of incomplete discharge. There is no dispute here that making certain polymers has been around for quite a long time. You can make them in a container, the problem is getting them out in a way that's useful and efficient. And the state of the art at the time, the solution at the time, was actually to use a piston and push this gel out. If you imagine jello, the last thing you want to do when you want to get jello out of the container is make the opening smaller. And so the prior art had two approaches to this, have no constriction in the opening, leave it wide open and let it fall out by its own weight—

Chief Judge Prost: Or that—

Mr. Riddle: Or, no, or use a piston and press it out so that you get all of it out. The '215 patent, is a very different situation, the '215 patent—

Judge Taranto: One second, can I just ask—in column 1 of the patent that's at issue here, the '329 patent, there is a long middle paragraph about JPA93/57181, some—a Japanese something patent application whatever. Doesn't that describe the use of inert gas to push polymer out of a reactor?

Mr. Riddle: So thank you for pointing that out. The JP '181 reference is a piston reference, which uses a piston. And in that reference, what the inventors in JP '181 said, is that if you just use inert gas, there are problems, and those problems include the gas breaking through the side of the reactor and the gel and escaping, in which case the gel remains trapped in the reactor so you can't get it out. This is the problem that the '329 was designed to address. Not being able to get the gel out of the reactor.

Judge Taranto: What about the section 103(c) argument your opposing counsel made. Did it not exist?

Mr. Riddle: So the 103(c) argument was never made in front of the PTAB Board. And it was a mistake in the briefing, but I would submit it's an honest mistake because both attorneys in this case made the mistake—

Chief Judge Prost: What mistake, how would you characterize the mistake? By relying on it at all? Is that what you're saying is the mistake? What is the mistake?

Mr. Riddle: The mistake is that 103(c) applying to 102(e) art—the effective date of that statute—both attorneys in this case in the IPR misunderstood the effective date of that 103(c) exception.

Judge Taranto: So it was raised below?

Mr. Riddle: No it was raised at the hearing. The PTAB put the question to SNF's counsel, why didn't you—the exact question—this panel has it, why didn't you raise the '215 patent earlier or another piece of art that was subsequent to the '215, the '948—why wasn't that raised in the petition, and SNF's counsel in front of the PTAB said, well, they could have easily sworn behind it because it was under 103(c). I think this was a mistake. It was a mistake in our briefing. I'm not trying to hide from that, but it wasn't an issue raised before the PTAB and it wasn't a basis for the PTAB's decision in any form.

Judge Taranto: What in the—what was the ground of the Board's treatment of the '597 prior art, starting with the claim construction that substantially all of the material has to be removed? What did it say about the '597?

Mr. Riddle: What the board said about the '597 is that neither the '597 taught the extent of removal from that reactor. Neither of the primary references, the '944 nor the '597, taught how much gel was removed from the reactor. And that's a natural consequence—

Judge Taranto: Well let me just, I guess, tell you what connection is in my mind and then you can tell me why—clarify things for me. It may be for me the most important aspect of the claim construction of the patent we have in front of us that a definite article "the," follows the "removal" word, not the "removal" word all by itself. The definite article suggest it's the entirety of the material that is being removed. Something different from the distinction between the word "discharge" and the word "remove." Mr. Dabney points out on column 3 of the '597, at line 70, that that piece of prior art also uses the word "the," that leaves me wondering why it doesn't have the same meaning that in effect undergirds the claim construction of the patent at issue here.

Mr. Riddle: I think there's two parts to the answer. Number one, in the '597 patent, the example at column 3 line 70 is just that, an example. It's not a claim. That's not claim language where you look at the antecedents the same way, you give those antecedents particular weight.

Judge Taranto: Why would that matter? It teaches everything that's in the document including in that example getting rid from the vessel of, let's say, the entirety of the material that was in the vessel.

Mr. Riddle: So, first of all, the '597 never mentions that it's trying to get rid of all of the material.

Judge Taranto: But if the word "the" has that fairly natural implication, and maybe you want to tell me that I'm reading too much into the word "the" and that the claim construction of the patent in front of us, the '329, doesn't really depend on that, but rather depends on a sense from the spec as a whole of the definition of the problem, that the problem that it was addressing was getting rid of all of it and now it's got something—it's got a solution to that, and so in the context of this patent, not particularly from the word "removed," which I must say is not to my mind very distinct from the word "discharge," but that when that word is being used in the context of this patent, it's not in reference simply to the action at any given moment but in specific reference to the ultimate end-point of the object of that word.

Mr. Riddle: If I understand your Honor, in the '597 patent, this example actually states, "the resultant gel-like polymer solution was discharged from the vessel." That's entirely consistent with the chemical engineering understanding of the word "discharge" being a passive phenomenon. You fill a reactor up. You want to drain it. Let's say it has water. I want to discharge it, I open the valve and gravity discharges it. That's not an active step of making sure everything is out of the reactor. That's a fundamental distinction between even the plain meaning of "discharge" and "removing."

Chief Judge Prost: I guess I'm struggling with that. I don't want to interrupt your train of thought, but all of the examples except for one—numerous examples in the '329 do not use the word "remove." They use the words "discharge without residue."

Mr. Riddle: You're right. In the examples they talk about, when they use the word discharge, there's always a qualifier with it. That's the point. When "discharge" is used, you have to qualify it because it's a passive action. So when you say "discharge," do you mean "just release some and residue is ok" or do you mean "get it all out"? So if you say discharge you have—

Chief Judge Prost: So "removal" is equal to "discharge without residue"?

Mr. Riddle: Yes. "Removal" is the same thing as "complete discharge." The plain dictionary definition as the PTAB noted for "removal" is "to eliminate, to get rid of," whereas "discharge" can just be "to pour out." Stuff can remain, and that's a fundamental—that's—what's important—because that's the fundamental purpose of this invention, was to address all these problems in the prior art of incomplete discharge.

Chief Judge Prost: Well going back to Judge Taranto's question then about line 70 of the '597 patent, "the resulting solution was discharged." You're saying that's something less than "without residue"? It has to explicitly talk about "without residue"? Is that what we're talking about?

Mr. Riddle: Well, I think the problem with the '597 patent is it says nothing about the extent of discharge, and this is a situation where because a reference says nothing, you can read into it as much or as little as you'd like, because the purpose of the '597 patent was downstream processing, so it has no information about the extent of discharge, it has no information about the purpose of discharge other than getting it to the next step—

Chief Judge Prost: Well, what about the '215, which explicitly calls out "discharged without residue by injecting inert gas"? What's—why isn't that sufficient?

Mr. Riddle: Well in the '215—I mean if you take those words in isolation—but that's not the reference. The reference is about a thermoplastic material, so first of all it's not the same kind of polymer, it's a multistage reactor process, where in those stages, gas pressure is used to move the material—and it's not purely a polymer gel in that it is—interposed within that thermoplastic are solid catalyst particles and there what is discharging—

Chief Judge Prost: But the Board makes the argument about the differentiation being the number 6, the discharge aid. Did you make that argument?

Mr. Riddle: Yes. The discharge aid—

Chief Judge Prost: Did you make that—you made that argument below to the Board?

Mr. Riddle: Yes.

Chief Judge Prost: Ok.

Mr. Riddle: That discharge aid, as the Board pointed out, was in the conical portion of the reactor. What the '215 patent teaches is that to discharge a material you can have a discharge aid in the conical portion of the reactor that can be either a piston, right, which is in the prior art, or a screw auger type thing where you can pull it down as a screw—pull the gel down. So the Board noted that the ’215 patent itself taught the use of a discharge aid within the reactor, and that was the fundamental distinction.

Chief Judge Prost: Isn’t 6 the discharge aid?

Mr. Riddle: Uh, yes, but—in the crude drawing, yes. But in the description—I mean this is not a drawing that’s to scale or is representative of the invention. It’s just schematically showing features in a rough sense.

Judge Taranto: What’s the portion of the ’215 that speaks of the discharge aid inside the vessel as opposed to six, seven? I think at column four, line 48.

Chief Judge Prost: What page in the appendix?

Judge Wallach: 1898.

Mr. Riddle: Appendix 1898. In there at about line 48 where the ’215 patent in the appendix 1898 states that “the discharge aid in the conical part of the reactor,” so—

Judge Taranto: I’m sorry, and the, what’s the—I think I’ve a little bit lost the point of why we’re talking about this?

Mr. Riddle: I think that actually is a good point because the ’215 patent’s not even a ground in the petition for—before the PTAB. The ’215 is this background knowledge argument.

Judge Taranto: The way the Board at least described it—it didn’t describe the ’215 as a freestanding piece of prior art but rather it was interpreting the 7 or 8 lines at the bottom of column one, top of column two and the ’329 patent which is at issue here and asking “what does that set of lines admit about the prior art?” And it was interpreting that language and it started with language that doesn’t actually refer to anything but the injection of inert gas and nevertheless saying, “well, we’re not quite sure what those words mean so we’re going to look at the ’215 prior art to understand the scope of the admission, and when we look at it we see that this scope of the admission is not something that covers the current claim as construed, because as we construe it we—we construe it to mean,” what?

Mr. Riddle: I think—

Judge Taranto: Inert air only doing the complete—the effectively complete removal?

Mr. Riddle: That’s part of it, but I think what the PTAB was saying was that you can’t look at the ’215 patent and learn from it that using a taper with inert gas would effect substantially complete discharge, because the ’215 patent was completely silent as to the benefit or function of a taper in terms of getting this substantially complete removal. So there was no teaching in the ’215 patent that would allow you to connect any dots to the grounds at issue. And I should say that the ’597 patent at issue did not teach a conical taper. That’s an extrapolation that the petitioner made below based on the sizes. Nowhere in the ’597 patent is there a teaching of a conical taper. There’s just—

Judge Taranto: I don’t remember the details but were there—was there a pair of diameters, one larger than the other?

Mr. Riddle: That’s correct. In the ’597—

Judge Taranto: I’m sorry, so what is not logically unavoidable when you have the diameter—small diameter vessel that’s— it’s not conical because it could be what?

Mr. Riddle: Because it could—all the ’597 patent teaches is that it has a bottom outlet. Bottom on the floor? Bottom on the side is possible. It could hemispherical, it could be flat. Those are all common shapes of reactors in chemical engineering. There is nothing in the ’597 patent to specifically state that this is a conical reactor.

Judge Wallach: In that ’215 the item 5 in that chart is referred to as a “displacement apparatus.” I agree with you, it doesn’t say that the discharge aid is not in the cone—in the conical section. But on the other hand, it does say that 5 “may be installed at the lower conical end.” But you are telling us that what’s labelled 5 there is in fact the discharge aid.

Mr. Riddle: With respect, I’m not saying that. What I —

Judge Wallach: You pointed to it when you said it!

Mr. Riddle: I am saying this area, this cone area.

Judge Wallach: Ok.

Mr. Riddle: 5, this disrupter mechanism, if you think about it—

Judge Wallach: I get what it is, but—

Mr. Riddle: Ok.

Judge Wallach: —so you’re saying that 6 is actually somewhere in the cone.

Mr. Riddle: According—

Judge Wallach: From the description it doesn’t say it’s not, it just doesn’t say.

Mr. Riddle: Well the description in the ’215 patent, actually line 48 on appendix 1898 column 4 of the ’215 patent, specifically says that the discharge aid is in the conical part of the reactor.

Judge Wallach: Hang on just—yeah you're right, you're right.

Mr. Riddle: So what I'm saying is that the '215 patent—what it teaches us—

Judge Wallach: Gotcha.

Mr. Riddle: —it teaches the location, and that is in the cone.

Judge Wallach: So your opposing counsel is incorrect in that argument then—in his analysis of that diagram?

Mr. Riddle: I believe so as that's what the PTAB found as well.

Judge Wallach: Uh-huh.

Mr. Riddle: I see my time is up.

Chief Judge Prost: Thank you.

Mr. Riddle: Thank you.

Chief Judge Prost: [inaudible]

Judge Wallach: You weren't able to see the chart, but you followed what I was asking about?

Mr. Dabney: Yes, and, and, and, and the words used were—

Judge Wallach: And how do you respond? How—wait, wait, don't talk. How do you respond to line 48 in column 4?

Mr. Dabney: Because what it's—let me say first of all, the—what we've been talking about here—there's been only one person who interpreted this who was skilled in the art. That was our expert. The question was raised, "did they raise this before the Board?" The answer is "they did not." Their expert did not address this at all because at that time, based on their erroneous, apparently, view of 103(c), they took the position this wasn't relevant, so their expert did not address it at all. So the evidence on what this patent disclosure means—

Judge Wallach: Can I read this?

Mr. Dabney: Yes, so getting to your point—

Judge Wallach: Thank you.

Mr. Dabney: I think what that line is saying is that it's forced continuously toward the discharge aid in the conical part, that is to say it's fully consistent with 6 correctly identifying the discharge aid is down here and it's pushing it towards the discharge aid in the conical portion so—

Judge Wallach: Wait a minute. It's not, from what you're saying, it's not forced in the cylindrical part, is that right? Is that what you're telling me?

Mr. Dabney: No no no no no—the inert gas is pushing down on the anhydrous gel—

Judge Wallach: Right.

Mr. Dabney: —and it's saying it's pushing it through the conical portion towards the discharge aid. That's the way our expert interpreted that.

Judge Wallach: So you read "in" as "through"?

Mr. Dabney: No, "towards the discharge aid in the conical part"—in other words, you could say it's—"in the conical part," is modifying "forced continuously in the conical part," towards the discharge aid. That's what our expert—that's how our expert interpreted that and that's consistent with how the figures are labeled in the drawing. Now, counsel stood here and said that the word "discharge" is only used with modifier. Not so. The abstract of the patent says, this is in page 825 of the appendix, the abstract says, "and discharge of the gelatinous reaction mixture from the reactor by injection of an inert gas." There again the word "discharge, ejecting, pushing out, removing," they're all describing an operation. And judge Taranto, even if we don't think this is the correct construction because in this patent the disclosure is "optimally the gel is there at an average constant level," so for a continuous process, which, this patent, in column 4 says is an embodiment—a continuous process embodiment—

Judge Taranto: Is there injecting the gel from the side?

Mr. Dabney: That—before this oral argument I'd never heard that—

Judge Taranto: Never mind, I'm sorry to—

Mr. Dabney: Yeah, no no no, but the point is if you interpret "removing" as more or less interchangeable with "discharging, ejecting, pushing out," as an action which will go on for whatever length of time the machine/system is causing that action to happen, well then you describe both batch processing—

Judge Taranto: Right but let me just ask—what do you do, well what's your response to the following characterization of the spec. The spec says, "there has been a problem in the prior art. There have been a variety of ways of getting stuff out. The inert gas way doesn't get it all out and we have a solution to that. So our claim really had better be about getting all of the stuff out—

Mr. Dabney: And, and, and—

Judge Wallach: —because that's the problem that we think we can solve, with inert gas. And we described in column 1 how the prior art doesn't solve that problem.

Mr. Dabney: For water soluble hydrous gels only, and that's what the examiner said. The—column 1, column 2 says there's no problem getting it all out in the '215. This is an anhydrous gel. What we've done here, and the reason our claims are limited to the hydrous gel is that was what they then said was their innovation—taking this and using this process for the hydrous gel polymer like poly-acrylamide, poly-acrylate. And that's what the statement of reasons for allowance said. It was only in the midst of litigation when they were confronted with evidence that that was not a new idea, this has been used for poly-acrylamide too, that they came up with this new theory that what the innovation here was not the idea of substituting a hydrous for an anhydrous monomer, but operating the system for long enough to achieve a state of substantial emptiness. And that is a strategy that makes sense in IPR because of the severe procedural constraints on the petitioner to try to respond to a new on the fly argument like that, but as your Honors pointed out, the word "the" is the Achilles' heel in that argument because the '597 patent very clearly, as our expert says, discloses discharge of substantially all, even under their construction. So in our view, the case clearly requires a remand because the proper interpretation of "removing" is—as a present participle verb—an action as opposed to an operating condition, and in the alternative, the Board's interpretation of this—now that we know that the whole patent owner presentation was based on an erroneous view of 103(c)—I think it's not accurate to say that this was not part of their presentation. It was built into their presentation, which is why this was never discussed by their expert. The board should not have, on its own, allowed the patent owner—

Judge Wallach: Did SNF argue below, as your opposing counsel says, on that very point?

Mr. Dabney: There was an—one of the things—what very point?

Judge Wallach: Your opposing counsel says that when the issue was raised by the Board, your counsel below said, "well they could just argue around it."

Mr. Dabney: Not true. '215 would be 102(b) prior art in any event. So that point is simply not true. It was another reference—

Judge Wallach: So it wasn't said, in other words.

Mr. Dabney: Not as to the '215. There was an error made as to something we haven't talked about at all here this morning, which was the '948 patent, but the '215 was always insisted upon and discussed at length in our expert submissions 1878 to 1882 of the JA that this is prior art and this shows that their expert was wrong when he said, quote, this is exhibit 2016 "Those who attempted to use inert gas to remover polymer gel from a reactor found that it was not a viable option." Their argument to the Board was not that this didn't work for hydrous gels, but that it didn't work at all. And that was untrue. And that was built on what we now have admitted on the record was an erroneous view of the law on which they erroneously didn't show this to their experts. So we thank very much the Court's attention and allowing me to go a little bit over.

Chief Judge Prost: Thank you. Thanks to both sides, and the case is submitted. That concludes our proceedings for today.

Clerk: All rise.

To whom it may concern:

This is to certify the attached transcription of English audio is an accurate representation from the media received by this office. This transcription is designated as:

Federal Circuit Argument

SNF Holding Company

v.

BASF Corporation

Appeal No. 16-2565

To the best of my knowledge the accompanying text is a true, full and accurate transcription of the specified audio.

Jennifer Flores

UNITED STATES COURT OF APPEALS FOR THE FEDERAL CIRCUIT

NOTICE OF ENTRY OF JUDGMENT WITHOUT OPINION

JUDGMENT ENTERED: 10/17/2017

The judgment of the court in your case was entered today pursuant to Rule 36. This Court affirmed the judgment or decision that was appealed. None of the relief sought in the appeal was granted. No opinion accompanied the judgment. The mandate will be issued in due course.

Information is also provided about petitions for rehearing and suggestions for rehearing en banc. The questions and answers are those frequently asked and answered by the Clerk's Office.

Costs are taxed against the appellants in favor of the appellee under Rule 39. The party entitled to costs is provided a bill of costs form and an instruction sheet with this notice.

The parties are encouraged to stipulate to the costs. A bill of costs will be presumed correct in the absence of a timely filed objection.

Costs are payable to the party awarded costs. If costs are awarded to the government, they should be paid to the Treasurer of the United States. Where costs are awarded against the government, payment should be made to the person(s) designated under the governing statutes, the court's orders, and the parties' written settlement agreements. In cases between private parties, payment should be made to counsel for the party awarded costs or, if the party is not represented by counsel, to the party pro se. Payment of costs should not be sent to the court. Costs should be paid promptly.

If the court also imposed monetary sanctions, they are payable to the opposing party unless the court's opinion provides otherwise. Sanctions should be paid in the same way as costs.

Regarding exhibits and visual aids: Your attention is directed to FRAP 34(g) which states that the clerk may destroy or dispose of the exhibits if counsel does not reclaim them within a reasonable time after the clerk gives notice to remove them. (The clerk deems a reasonable time to be 15 days from the date the final mandate is issued.)

FOR THE COURT

/s/ Peter R. Marksteiner
Peter R. Marksteiner
Clerk of Court

16-2565 - SNF Holding Company v. BASF Corporation
United States Patent and Trademark Office, Case No. IPR2015-00600

NOTE: This disposition is nonprecedential.

United States Court of Appeals for the Federal Circuit

SNF HOLDING COMPANY, FLOPAM INC., CHEMTALL, INC., SNF SAS, SNF (CHINA) FLOCCULANT CO. LTD.,
Appellants

v.

BASF CORPORATION,
Appellee

2016-2565

Appeal from the United States Patent and Trademark Office, Patent Trial and Appeal Board in No. IPR2015-00600.

JUDGMENT

JAMES W. DABNEY, Hughes Hubbard & Reed LLP, New York, NY, argued for appellants. Also represented by JAMES W. BRADY, JR., JONATHAN W.S. ENGLAND, CHARLES J. MONTERIO, JR., Blank Rome LLP, Washington, DC.

ROBERT RIDDLE, Reed Smith LLP, Houston, TX, argued for appellee. Also represented by MATTHEW S.

GIBSON; JAMES CHRISTOPHER MARTIN, Pittsburgh, PA; SCOTT D. BAKER, San Francisco, CA.

THIS CAUSE having been heard and considered, it is

ORDERED and ADJUDGED:

PER CURIAM (PROST, *Chief Judge,* WALLACH and TARANTO, *Circuit Judges*).

AFFIRMED. *See* Fed. Cir. R. 36.

ENTERED BY ORDER OF THE COURT

October 17, 2017	/s/ Peter R. Marksteiner
Date	Peter R. Marksteiner Clerk of Court

United States Court of Appeals
for the Federal Circuit

CERTIFICATE OF SERVICE

I, Robyn Cocho, being duly sworn according to law and being over the age of 18, upon my oath depose and say that:

Counsel Press was retained by HUGHES HUBBARD & REED, Attorneys for Appellants to print this document. I am an employee of Counsel Press.

On **November 16, 2017**, Counsel for Appellant has authorized me to electronically file the foregoing **Petition for Rehearing and Rehearing En Banc** with the Clerk of Court using the CM/ECF System, which will send notice of such filing to the following registered CM/ECF users:

Robert Riddle
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In addition, 18 paper copies will be filed with the Court, via Federal Express, on the same date as above.

/s/ Robyn Cocho
Counsel Press

CERTIFICATE OF COMPLIANCE WITH TYPE-VOLUME LIMITATION, TYPEFACE REQUIREMENTS AND TYPE STYLE REQUIREMENTS

1. This petition complies with the type-volume limitation of Federal Rule of Appellate Procedure 35(b)(2)(A) or Federal Rule of Appellate Procedure 40(b).

The petition contains 3,639 words, excluding the parts of the petition exempted by Rule.

2. This petition complies with the typeface requirements of Federal Rule of Appellate Procedure 32.

__x___ The petition has been prepared in a proportionally spaced typeface using Times New Roman in a 14 point font or

____ The petition has been prepared in a monospaced typeface using ____________ in a ___ characters per inch_________ font.

/S/ JAMES W. DABNEY
James W. Dabney
Counsel for Appellants