US005891982A

United States Patent [19]

Graham

[11] Patent Number: 5,891,982

[45] Date of Patent: Apr. 6, 1999

[54] **CONTINUOUS PROCESS FOR PREPARING POLYESTER COPOLYMER RESIN**

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[21] Appl. No.: **744,768**

[22] Filed: **Nov. 6, 1996**

Related U.S. Application Data

[60] Provisional application No. 60/007,304, Nov. 6, 1995.

[51] **Int. Cl.** [6] **C08G 64/00**

[52] **U.S. Cl.** **528/176**; 528/190; 528/193; 528/194; 528/271; 528/272; 528/499; 528/501

[58] **Field of Search** 528/176, 190, 528/193, 194, 271, 272, 499, 501

[56] **References Cited**

U.S. PATENT DOCUMENTS

4,943,602 7/1990 Brueckmann et al. 524/600

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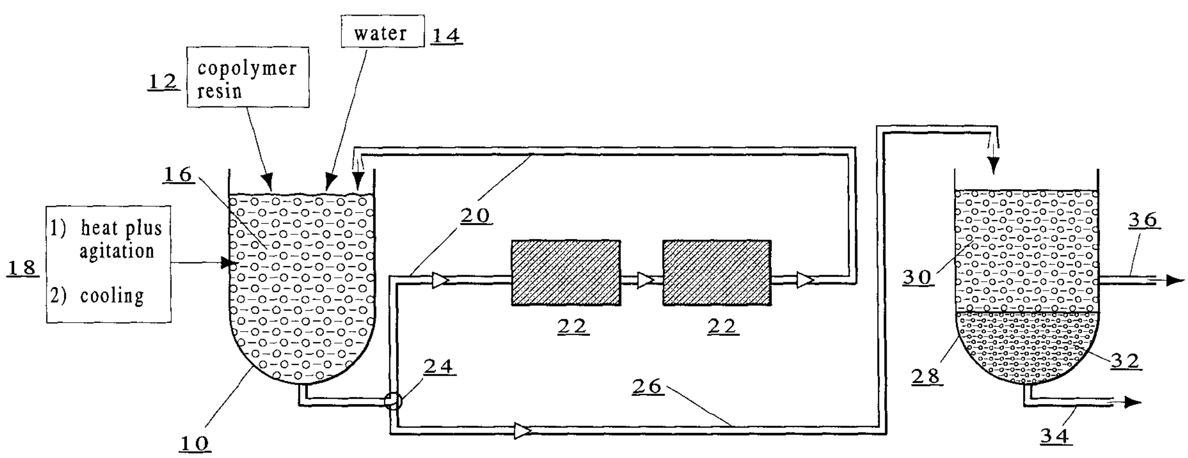
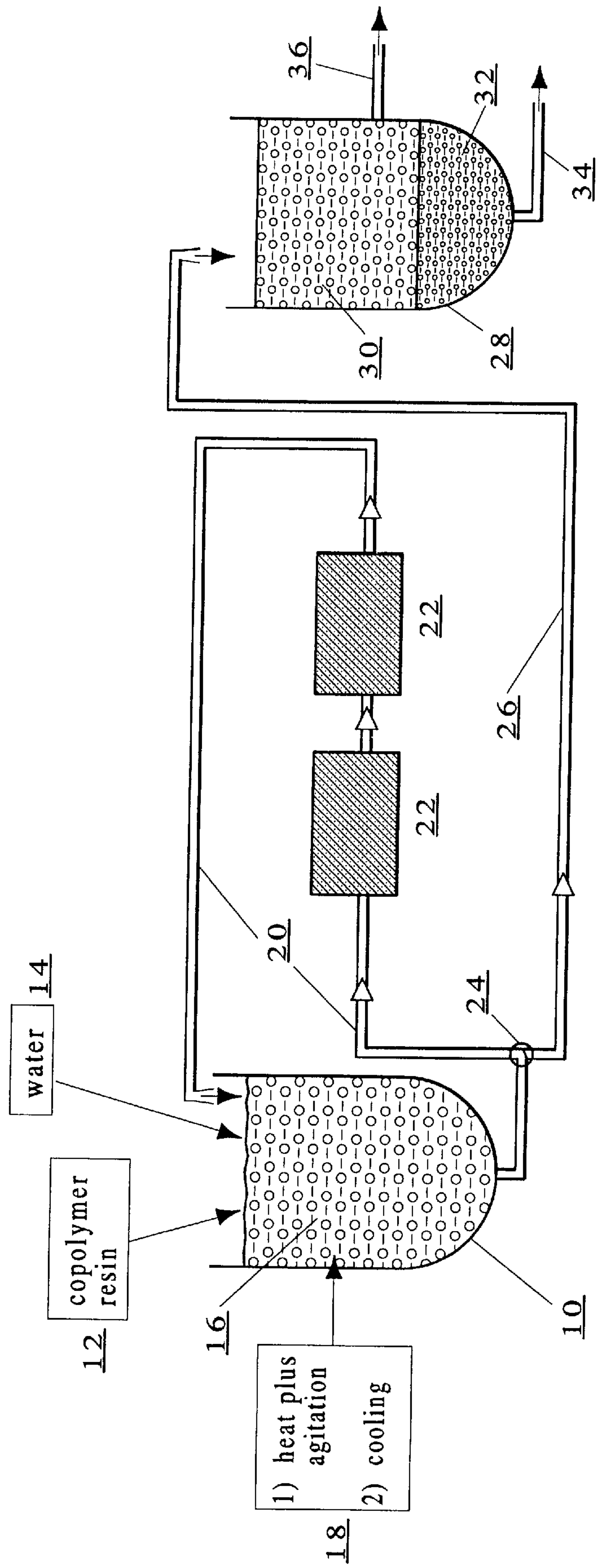
Attorney, Agent, or Firm—Michael I. Kroll

[57] **ABSTRACT**

The present invention relates to a process for preparing a dispersion of high grade polyester copolymer resin in water comprising 1) combining a crude polyester copolymer resin, having particles of various sizes, with water; 2) heating and agitating the resin/water mixture; 3) cooling the mixture; 4) continuously filtering the mixture to remove the largest size resin particles; 5) allowing the mixture to stand undisturbed so that any undispersed particles settle out as a sediment; and 6) removing the sediment, leaving a dispersion of the smallest resin particles in water. By using a plurality of filters and sequentially finer filter mesh sizes, a high grade product is attained.

25 Claims, 7 Drawing Sheets





36
32
34
30
28
22
22
26
20
24
water
14
copolymer resin
12
16
10
1) heat plus agitation
2) cooling
18

Fig. 1

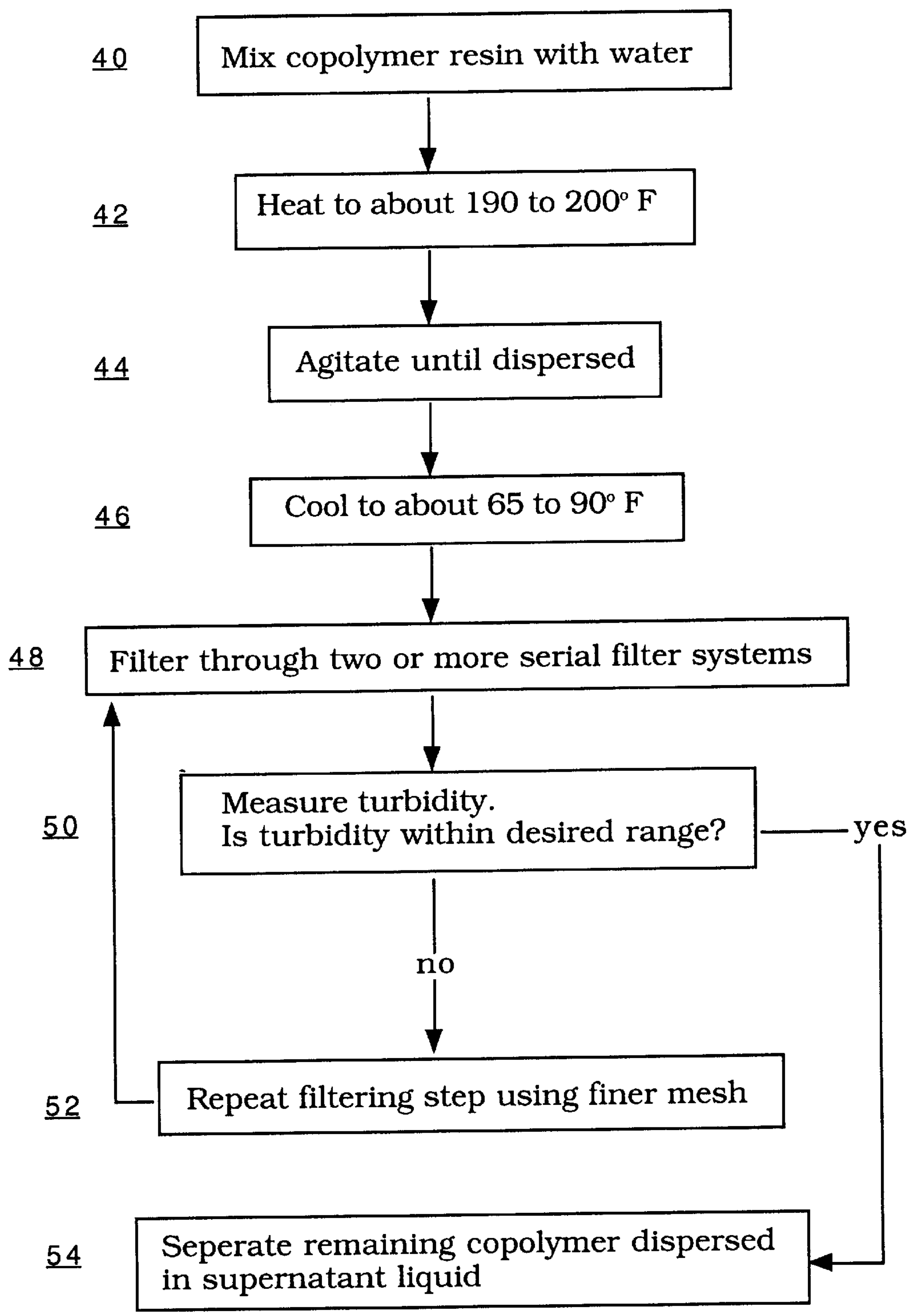

40
Mix copolymer resin with water
42
Heat to about 190 to 200° F
44
Agitate until dispersed
46
Cool to about 65 to 90° F
48
Filter through two or more serial filter systems
50
Measure turbidity.
Is turbidity within desired range?
yes
no
52
Repeat filtering step using finer mesh
54
Seperate remaining copolymer dispersed in supernatant liquid

Fig. 2

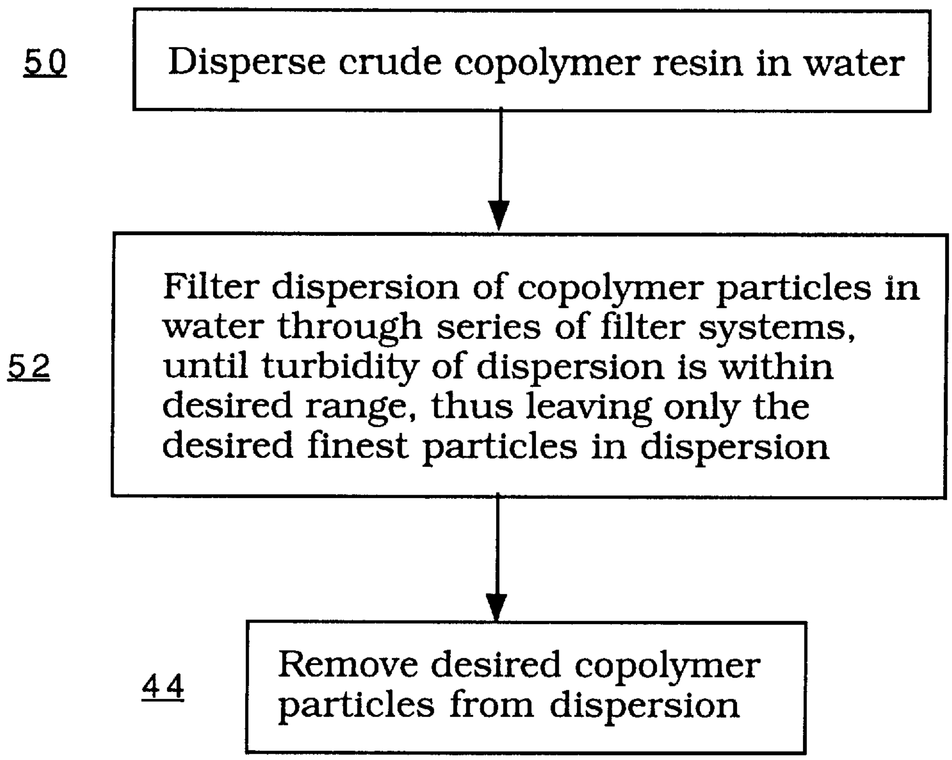

50
Disperse crude copolymer resin in water
52
Filter dispersion of copolymer particles in water through series of filter systems, until turbidity of dispersion is within desired range, thus leaving only the desired finest particles in dispersion
44
Remove desired copolymer particles from dispersion

Fig. 3

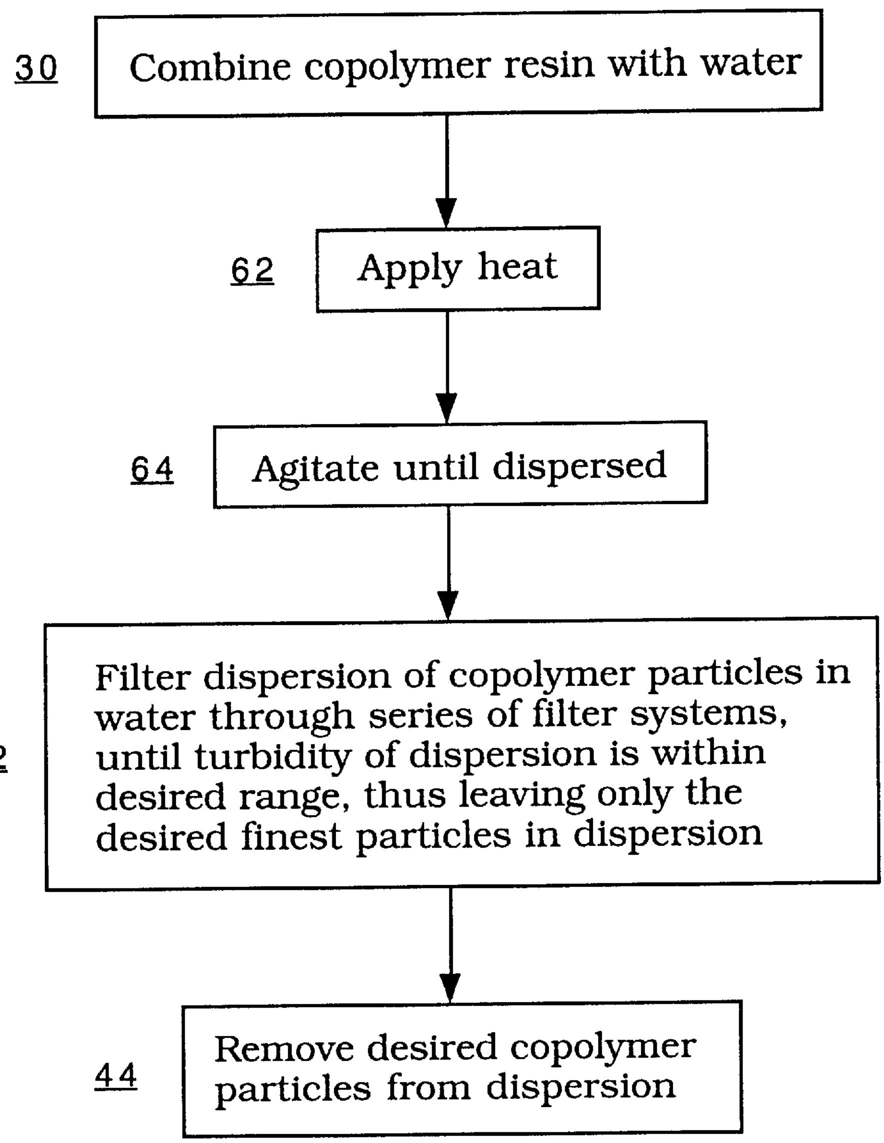

30
Combine copolymer resin with water
62
Apply heat
64
Agitate until dispersed
52
Filter dispersion of copolymer particles in water through series of filter systems, until turbidity of dispersion is within desired range, thus leaving only the desired finest particles in dispersion
44
Remove desired copolymer particles from dispersion

Fig. 4

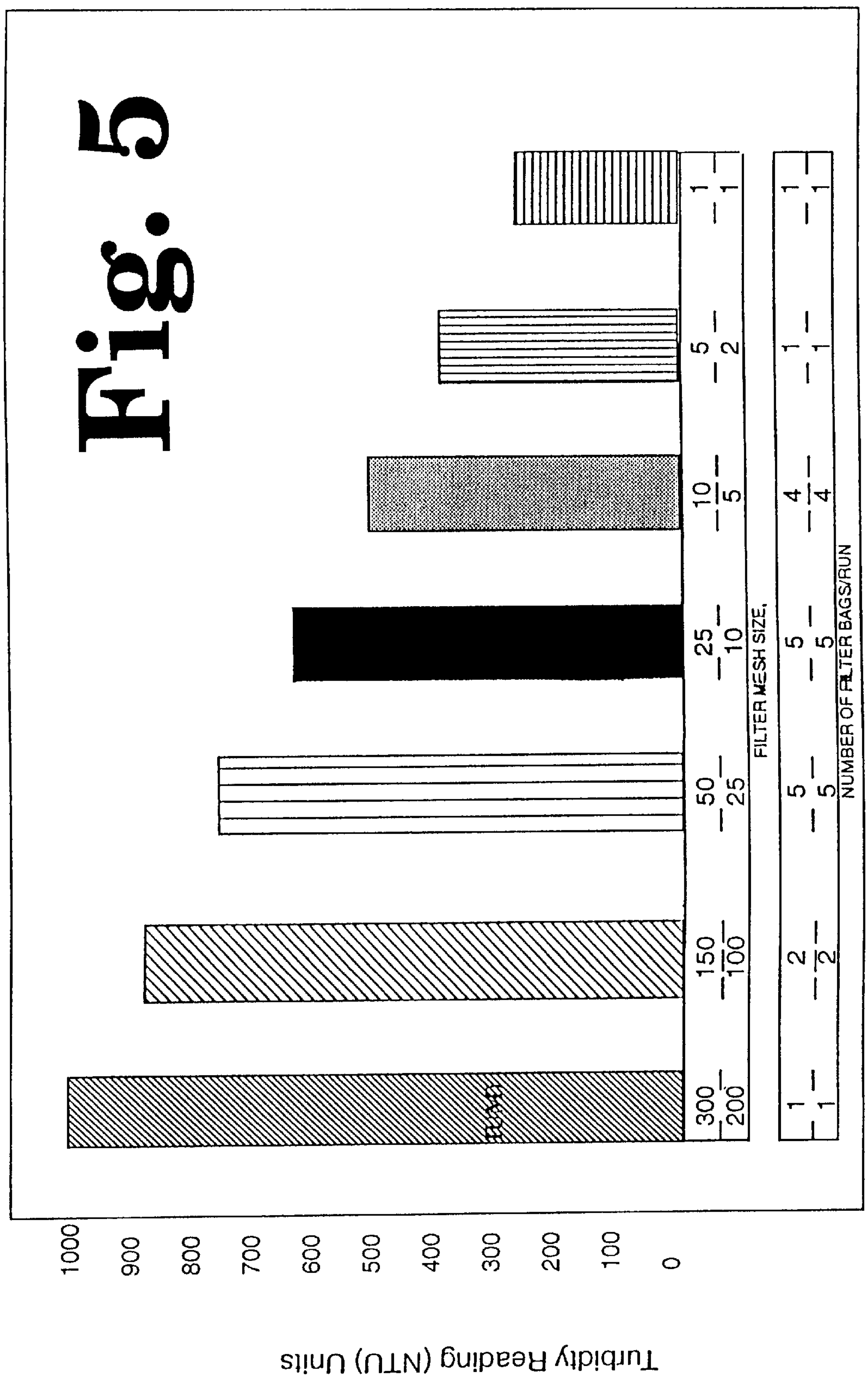

Turbidity Reading (NTU) Units
1000
900
800
700
600
500
400
300
200
100
0
300/200
150/100
50/25
25/10
10/5
5/2
1/1
FILTER MESH SIZE
1/1
2/2
5/5
5/5
4/4
1/1
1/1
NUMBER OF FILTER BAGS/RUN

Fig. 5

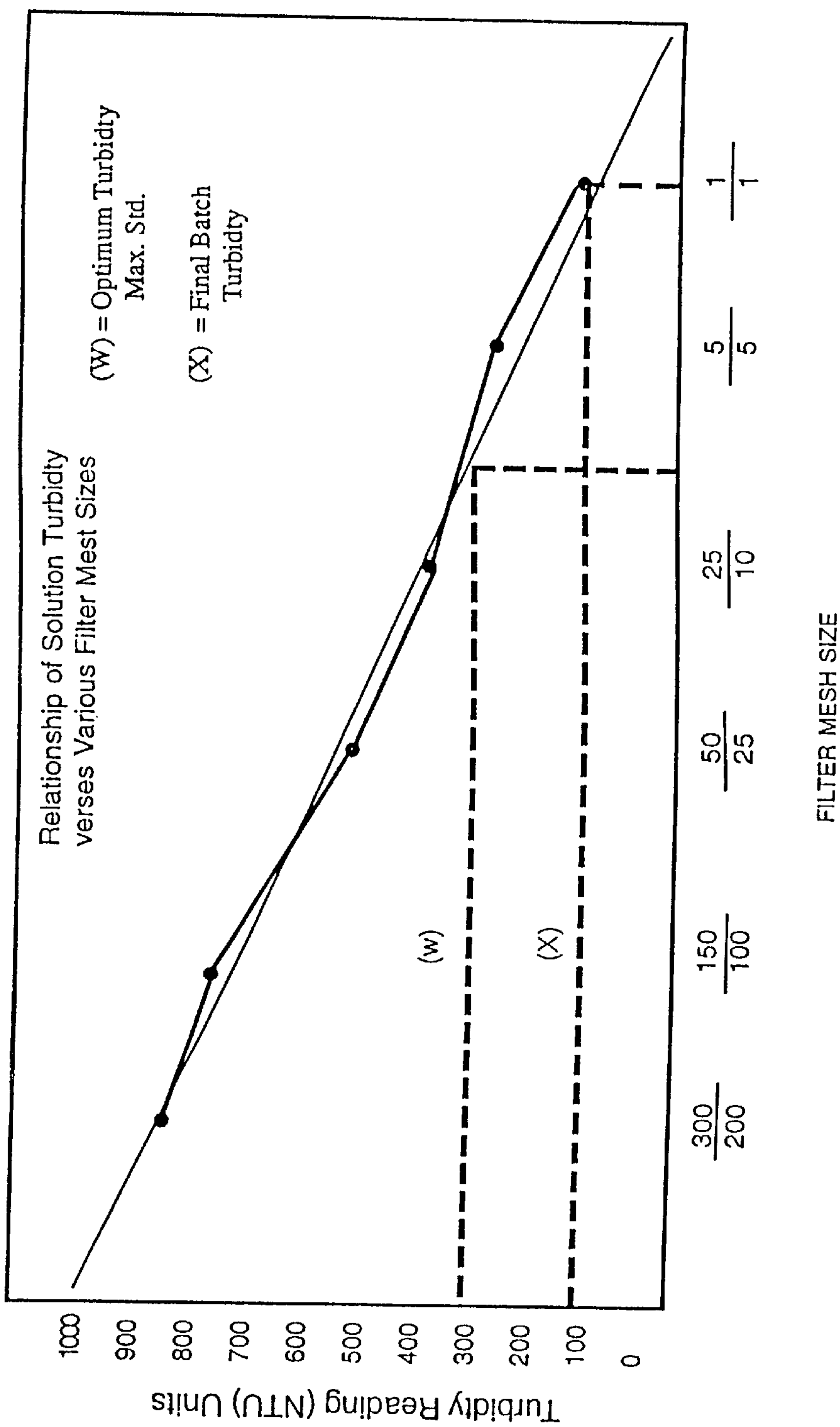

Relationship of Solution Turbidty
verses Various Filter Mest Sizes
(W) = Optimum Turbidty
Max. Std.
(X) = Final Batch
Turbidty
(w)
(X)
Turbidity Reading (NTU) Units
1000
900
800
700
600
500
400
300
200
100
0
300/200
150/100
50/25
25/10
5/5
1/1
FILTER MESH SIZE

Fig. 6

Property	Specification	Batch Number						
		1	2	3	4	5	6	7
Total Solid (%)	12.0-15.0	13.0	13.05	12.9	13.1	12.85	13.2	13.0
PH (5%) Solution	6.3-7.5	6.4	6.5	6.4	6.3	6.3	6.4	6.4
Specific Gravity (25%)	1.02 - 1.04	1.031	1.029	1.030	1.032	1.029	1.030	1.031
Turbidity (2% Solution)	300 NTU Maxium	12.0	100	105	85	110	105	130

Fig. 7

CONTINUOUS PROCESS FOR PREPARING POLYESTER COPOLYMER RESIN

CROSS-REFERENCE TO RELATED APPLICATION

This application derives from and claims the benefit of U.S. Provisional application Ser. No. 60/007,304, filed on 6 Nov., 1995.

BACKGROUND OF THE INVENTION

1. Field of the Invention

The instant invention relates generally to processes for preparing a polyester copolymer resin and more specifically it relates to a process for dispersing a crude polyester copolymer resin and separating out, via continuous multi-stage filtration, the smallest size particles present in the crude starting material.

2. Description of the Prior Art

It is known in the art to utilize filter systems to separate polyester copolymer resin dispersions. Speaking generally, these methods use inert filter aides such as diatomaceous earth. The most common filter press design consists of alternate plates and frames hung on a rack and forced tightly together with a hydraulic closing mechanism. Feed slurry is then pumped to the press under pressure. As the filtration proceeds, filter cakes build up on the filter cloths until the cakes form a nearly solid mass, requiring that they be removed for further processing. With these processes, the finished product (the polyester resin) is obtained in the form of the filter cake removed from the filter press, and the filtrate solution is the by-product. In the instant invention, however, the filtrate is the desired product and the removed sediment is the by-product.

SUMMARY OF THE INVENTION

The present invention is concerned with a process for preparing a dispersion of high grade polyester copolymer resin in water comprising 1) combining a crude polyester copolymer resin, having particles of various sizes, with water; 2) heating and agitating the resin/water mixture; 3) cooling the mixture; 4) continuously filtering the mixture to remove the largest size resin particles; 5) allowing the mixture to stand undisturbed so that any undispersed particles settle out as a sediment; and 6) removing the sediment, leaving a dispersion of the smallest resin particles in water. By using a plurality of filters and sequentially finer filter mesh sizes, a high grade product is attained.

A primary object of the present invention is to provide a process for preparing a dispersion of high grade polyester copolymer resin in water.

Another object of the present invention is to provide a process for preparing a dispersion of high grade polyester copolymer resin in water via a continuous serial filtration system.

An additional object of the present invention is to provide a process for preparing a dispersion of high grade polyester copolymer resin in water, which resin is suitable for use in manufacturing PET films.

A further object of the present invention is to provide a process for preparing a dispersion of high grade polyester copolymer resin in water which is environmentally friendly.

A still further object of the present invention is to provide a process for preparing a dispersion of high grade polyester copolymer resin in water that is easy and economical to implement using existing machinery.

The foregoing and other objects, advantages and characterizing features will become apparent from the following description of certain illustrative embodiments of the invention.

The novel features which are considered characteristic for the invention are set forth in the appended claims. The invention itself, however, both as to its construction and its method of operation, together with additional objects and advantages thereof, will be best understood from the following description of the specific embodiments when read and understood in connection with the accompanying drawings. Attention is called to the fact, however, that the drawings are illustrative only, and that changes may be made in the specific construction illustrated and described within the scope of the appended claims.

BRIEF DESCRIPTION OF THE DRAWING FIGURES

Various other objects, features and attendant advantages of the present invention will become more fully appreciated as the same becomes better understood when considered in conjunction with the accompanying drawings, in which like reference characters designate the same or similar parts throughout the several views.

FIG. **1** is a diagrammatic view of an apparatus for practicing the process of the present invention.

FIG. **2** is a flowchart illustrating the preferred, detailed steps of the process of the present invention.

FIG. **3** is a flowchart illustrating, in general terms, the steps of the process of the present invention.

FIG. **4** is a flowchart illustrating, in intermediately detailed terms, the steps of the process of the present invention.

FIG. **5** is a bar graph illustrating the relationship between the turbidity of the polymer/water mixture and the filter sequence being utilized.

FIG. **6** is a line graph illustrating the relationship between the turbidity of the polymer/water mixture and the filter sequence being utilized.

FIG. **7** is a table showing the various physical and chemical characteristics for the final water/polymer mixture after being processed according to the present invention, for a variety of different batches.

LIST OF REFERENCE NUMERALS UTILIZED IN THE DRAWINGS

10 mixing kettle for dispersing copolymer resin **12** in water **14**

12 crude copolymer resin from which an extremely fine, high quality resin is to be extracted

14 water

16 dispersion of resin **12** in water **14**, contained in the mixing kettle **10**

18 application of heat plus agitation to the resin **12** and water **14** mixture within mixing kettle **10** acts to create dispersion **16**, which is then cooled prior to filtering

20 conduits through which the cooled dispersion **16** is pumped to and from the filter system **22**

22 individual sequential filter in filter system

24 switch valve for directing the dispersion **16** either to the filter system **22** or to a settling tank **28**

26 conduit through which the dispersion **16** is pumped to the settling tank **28**

28 settling tank for allowing any undispersed particles **32** to settle out of the dispersion **16**

30 final dispersion which contains the desired fine copolymer particles
32 undispersed particles which settle to bottom of the settling tank 28
34 conduit for removing settled, undispersed particles 32 from the desired dispersion 30
36 conduit for drawing off the desired dispersion 30
40 initially, the crude copolymer resin 12 is combined with water 14 in the mixing kettle 10
42 the resin 12 and water 14 mixture is heated, preferably to a temperature of from about 180° to 200° F.
44 the resin 12 and water 14 mixture is thoroughly agitated in order create dispersion 16
46 the dispersion 16 is then cooled, preferably to a temperature of from about 65° to 90° F.
48 the cooled dispersion 16 is then filtered through a sequential filter system 22 in order to filter out the larger, unwanted particles of resin 12
50 the turbidity of the resultant dispersion is measured to determine if it is within desired guidelines; the less turbid the dispersion, the finer the particle size of the resin within the dispersion
52 if the turbidity is higher than desired, filtering is continued, either with the same filter system, or through a filter system of finer mesh
54 if the turbidity is within the desired range, the particle size of the resin within the dispersion is acceptable and the remaining resin in dispersion is separated from the supernatant liquid and packaged for use
60 the initial step of the process is to produce a dispersion of the crude copolymer resin in water
62 the dispersion of resin in water is filtered until the dispersion achieves a predetermined turbidity
72 the mixture of resin in water is heated to assist dispersion
74 the mixture of resin in water is agitated to assist dispersion

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Turning now descriptively to the drawings, in which similar reference characters denote similar elements throughout the several views, the Figures illustrate a process for for preparing a dispersion of high grade polyester copolymer resin in water.

As shown diagrammatically in FIG. **1**, and as illustrated in FIGS. **2** through **4**, the process of the present invention begins with a crude polyester copolymer resin. By "crude," it is meant that the resin, produced via conventional polymerization methods, is made up of polymer particles which vary greatly in size. There are some applications, however, which require that the polymer particles be of the smallest size only. Such applications include, for example, the manufacture of high-grade polyester PET film.

In the first step **40**, the crude copolymer resin **12** is mixed with an appropriate amount of water **14**. For example, a typical range is from about 10 to about 20% solids, more commonly about 12 to about 15% solids.

Next, the mixture **16** of resin **12** and water **14** is heated **42** and agitated thoroughly **44** in order to disperse the dispersible polymer in the mixture. It is expected that a temperature in the range of about 180° to about 200° F. will be sufficient for this. With regard to the agitation, there is no specific requirement, but a high speed mixing for one to two hours generally proves adequate.

Prior to filtering, it is preferred to cool the polymer/water mixture **16**, generally to a temperature of from about 65° to about 90° F. **46**, although some variance from this range will not significantly affect the efficiency of the process.

At this point, the mixture **16** is filtered **48** through a continuous serial filter system **22**. The filtering system preferred in the process of the present invention consists of a combination of liquid filter bags at various mesh sizes. It is preferred to use a plurality of filter systems, each capable of containing a plurality of filter bags. In its most preferred embodiment, the process utilizes two filter systems, each capable of containing from 1 to at least 5 filter bags. The mixture **16** is circulated, in a continuous manner, through the filter system, thus removing the copolymer particles large enough to be trapped within the filter bags. With regard to the filter bag mesh size, the first filter system in the process will generally have a mesh size of at least 200 microns.

Over the course of the filtration, the filter bags are preferably changed at periodic intervals, with smaller mesh sizes being used in each subsequent filtration. In addition, it has been found that the process will proceed more efficiently when each filter system in the series contains a filter or filters having a smaller mesh size than the one preceding it. For example, when two filter systems are connected in series, it has been found that, for the initial filter sequence, a first filter having a single filter bag of 300 micron mesh size and a second filter having a single filter bag of 200 micron mesh size can be effectively employed. When two filter systems are utilized, it is preferred that the second filter system utilize filter bags having a mesh size the same as or smaller than the first filter system. As a general guideline, the ratio of the mesh size of the first filter system to the second filter system will most often be between 1:1 and 5:1.

Eventually, these initial filters will have removed all or nearly all of the largest copolymer particles from the mixture, so replacement of the filter bags with smaller mesh size filter bags is in order. For example, in the above instance, it would be appropriate to replace the single 300 and 200 micron filter bags with dual 150 and 100 micron mesh size filter bags, respectively. With regard to the filter bag mesh sizes before and after a filter bag change, it is anticipated that a ratio of the second filter system mesh size before a filter bag change is made to the first filter system mesh size after a filter bag change is made will generally be from 1:1 to 5:1. For example, if a filter sequence is using two filter systems, a first with filter bags of 150 micron mesh size and a second with filter bags of 100 micron mesh size, then after a filter change, the first filter system would most often have filter bags with a mesh size of from 100 microns to 20 microns.

This process, of using progressively smaller mesh size filter bags,can be repeated, for example, down to 1 micron or smaller mesh size. Use of multiple bags in each filter systems improves the efficiency of the process. For example, the intermediate mesh sizes (from about 5 to about 50 microns) can effectively employ up to 5 or more filter bags per unit.

The progress of the filtration can be monitored, for example, by measuring the turbidity **50** of the mixture. As the larger particles are removed by the filtration, the turbidity decreases accordingly. For example, the initial copolymer/water mixture will often have an initial turbidity of about 1000 NTU or more, while the final product, after filtering with filter bags from 300 to 1 micron, will often yield a dispersion having a turbidity of 120 NTU or less. It is generally expected that a turbidity value of under 120 NTU will provide a copolymer resin of suitable quality. Accordingly, the turbidity is measured at various intervals.

If the turbidity is higher than desired and remains the same or nearly the same over consecutive measurements, then the filter bags should be changed to a smaller mesh size and the filtration continued. Once the turbidity reaches the desired level, however, the resulting dispersion contains copolymer particles of an acceptable size.

Optionally, the mixture is then transferred to a settling tank **28**, so that any undispersed particles can settle out of solution, forming a sediment **32**, which can easily be drained off the bottom of the settling tank, leaving only the desired dispersion **30**. Due to the continuous nature of the filtration, it can be readily appreciated that some large particles might not have been sent through the filtration system. This sedimentation will allow these particles to be readily removed.

EXAMPLE 1

Initially, 12–15% of the polyester copolymer resin is slowly added to a reaction kettle containing water at a temperature of 195°–200° F. and mixed at high speed for 1½ to 2 hours. After cooling, the mixture is filtered, using series filtration (two units) as shown in Table 1, below, with filter bags varying in mesh size. After a total of about 14 hours of filtering (each filtering run lasting from about 1½ hours to 3½ hours) with filters ranging in micron mesh size from 300 to 1 micron, a finish product with a final turbidity reading of 120 NTU is yielded.

TABLE 1

Filter 1		Filter 2		Ending Turbidity
Micron Size	# of Bags	Micron Size	# of Bags	(NTU)
(initial mixture prior to filtering)				~1000
300	1	200	1	~850
150	2	100	2	~780
50	5	25	5	~500
25	5	10	5	~380
5	2	5	2	~280
1	1	1	1	~120

It will be understood that each of the elements described above, or two or more together, may also find a useful application in other types of applications differing from the type described above.

While the invention has been illustrated and described as embodied in a process for preparing a dispersion of polyester copolymer resin in water, it is not intended to be limited to the details shown, since it will be understood that various omissions, modifications, substitutions and changes in the forms and details of the formulation illustrated and in its operation can be made by those skilled in the art without departing in any way from the spirit of the present invention.

Without further analysis, the foregoing will so fully reveal the gist of the present invention that others can, by applying current knowledge, readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of this invention.

What is claimed as new and desired to be protected by letters patent is set forth in the appended claims.

I claim:

1. A process for preparing a dispersion of polyester copolymer resin in water comprising the steps:

a) combining a crude polyester copolymer resin, having particles of various sizes, with water;

b) heating said resin/water mixture;

c) agitating said resin/water mixture;

d) cooling said mixture;

e) continuously filtering said mixture through a plurality of filter units, each of said filter units containing 1 or more filter bags, to remove the largest size resin particles from said mixture;

f) transferring said mixture to a settling vessel;

g) allowing said mixture to stand undisturbed so that any undispersed particles in said mixture settle out as a sediment, and

h) removing said sediment from said mixture, leaving a dispersion of the smallest resin particles in water, wherein said mixture is heated to a temperature of from about 180° to about 200° F. in said heating step.

2. A process as defined in claim **1**, wherein said mixture is cooled to a temperature of from about 65° to about 90° F. in said cooling step.

3. A process as defined in claim **2**, further comprising the step of measuring the turbidity of the mixture during the filtering process, transferring the mixture to the settling vessel when the turbidity of the mixture falls below a predetermined level.

4. A process as defined in claim **3**, wherein each of said filter units contains from 1 to 5 filter bags.

5. A process as defined in claim **4**, comprising a first and a second filter unit arranged in series.

6. A process as defined in claim **5**, wherein said first filter unit contains from 1 to 5 filter bags having a first mesh size and said second filter unit contains from 1 to 5 filter bags having a second mesh size, wherein said second mesh size is the same as or smaller than said first mesh size.

7. A process as defined in claim **6**, wherein the ratio of said first mesh size to said second mesh size is from 1:1 to 5:1.

8. A process as defined in claim **7**, wherein said filtering step further comprises periodically changing said filter bags in said filter units so that said filtering step utilizes an initial filter bag mesh size for each filter unit, a final filter bag mesh size for each filter unit, and a plurality of intermediate filter bag mesh sizes for each filter unit.

9. A process as defined in claim **8**, wherein said final filter bag mesh size for said second filter unit is 1 micron or less.

10. A process as defined in claim **9**, wherein said initial filter bag mesh size for said first filter unit is 200 microns or greater.

11. A process as defined in claim **10**, wherein the ratio of the second mesh size before a filter bag change is made to the first mesh size after a filter bag change is made is from 1:1 to 5:1.

12. A process for preparing a dispersion of polyester copolymer resin in water comprising the steps:

a) combining a crude polyester copolymer resin, having particles of various sizes, with water;

b) heating said resin/water mixture;

c) agitating said resin/water mixture;

d) cooling said mixture; and

e) continuously filtering said mixture through a plurality of filter units, each of said filter units containing 1 or more filter bags, to remove the largest size resin particles from said mixture, wherein said mixture is heated to a temperature of from about 180° to about 200° F. in said heating step.

13. A process as defined in claim **12**, wherein said mixture is cooled to a temperature of from about 65° to about 90° F. in said cooling step.

14. A process as defined in claim **12**, further comprising the step of measuring the turbidity of the mixture during the filtering process, transferring the mixture to the settling vessel when the turbidity of the mixture falls below a predetermined level.

15. A process as defined in claim **14**, wherein each of said filter units contains from 1 to 5 filter bags.

16. A process as defined in claim **15**, comprising a first and a second filter unit arranged in series.

17. A process as defined in claim **16**, wherein said first filter unit contains from 1 to 5 filter bags having a first mesh size and said second filter unit contains from 1 to 5 filter bags having a second mesh size, wherein said second mesh size is the same as or smaller than said first mesh size.

18. A process as defined in claim **17**, wherein the ratio of said first mesh size to said second mesh size is from 1:1 to 5:1.

19. A process as defined in claim **18**, wherein said filtering step further comprises periodically changing said filter bags in said filter units so that said filtering step utilizes an initial filter bag mesh size for each filter unit, a final filter bag mesh size for each filter unit, and a plurality of intermediate filter bag mesh sizes for each filter unit.

20. A process as defined in claim **19**, wherein said final filter bag mesh size for said second filter unit is 1 micron or less.

21. A process as defined in claim **20**, wherein said initial filter bag mesh size for said first filter unit is 200 microns or greater.

22. A process as defined in claim **21**, wherein the ratio of the second mesh size before a filter bag change is made to the first mesh size after a filter bag change is made is from 1:1 to 5:1.

23. A process as defined in claim **22**, further comprising the steps:

f) transferring said mixture to a settling vessel;

g) allowing said mixture to stand undisturbed so that any undispersed particles in said mixture settle out as a sediment; and

h) removing said sediment from said mixture, leaving a dispersion of the smallest resin particles in water.

24. A dispersion of polyester copolymer resin in water produced by the process defined in claim **1**.

25. A dispersion of polyester copolymer resin in water produced by the process defined in claim **12**.

* * * * *