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(54) Title: A PROCESS FOR CONVERSION OF GREY ACRYLIC FIBER WASTE TO ACRYLIC POWDER

(57) Abstract: The invention discloses a process for conversion of grey acrylic fiber waste to acrylic powder comprising of the steps of (a) dissolving the acrylic wastes in a solvent in the concentration of 1-25% and temperature of not more than 90°C; (b) filtration through any coarse filter media to remove any un-dissolved and other impurities before precipitation; (c) precipitation of acrylic polymer from the solution using any commercially available primary-, secondary- and tertiary alcohols in the ratio of 1:1 to 1:10 volume by volume of polymer solution and alcohols; (d) vacuum filtration to remove the solvents mixture; (e) dispersing the polymer in hot/cold DM water in the ratio of 1:5 to 1:20 weight by volume to remove residual solvents; (e) pelletizing the wet precipitate before drying or directly drying the polymer at elevated temperature to remove moisture; (f) pulverizing the dried polymer and sieving to obtain fine acrylic powder; (g) recovering the used solvents by distillation process.

A PROCESS FOR CONVERSION OF GREY ACRYLIC FIBER WASTE TO ACRYLIC POWDER

FIELD OF INVENTION:

The present invention relates to a novel process for converting grey acrylic fiber waste to acrylic powder which can be used in acrylic fiber production line without affecting the properties of virgin acrylic fiber.

BACKGROUND OF INVENTION:

Acrylic polymer is widely formed by free-radical polymerization in aqueous suspension. The acrylic polymer formed is purified by series of filtration and washing sequence with DM water, dried and pulverized for fiber production. The fiber is produced by dissolving the polymer in solvents such as N,N-dimethylformamide (DMF); dimethyl sulfoxide (DMSO); aqueous sodium thiocyanate (NaSCN) or dimethylacetamide (DMAc) in a precise concentration along with other additives; metering it through a multi-hole spinnerets and coagulating the resultant filaments in an aqueous solution of the same solvent (wet spinning) or evaporating the solvent in a stream of heated inert gas (dry spinning). This follows by washing, stretching, drying and crimping to form acrylic tow and staples fiber.

The acrylic fiber wastes are generated in different stages of fiber production line in the form of acrylic soft waste, acrylic tangled tow waste, acrylic off grade soft and hard waste etc. These

wastes find their market in Asian countries, where they are subjected to manual separation and production of yarn either in the actual form or blending with other fibers. The acrylic fiber wastes also find application in manufacturing of acrylic base adhesives and paints.

The global demands for synthetic fiber and higher cost of acrylic fibers in compare to polyester fibers require measures to minimize the waste and reduce the production cost.

Considering the need for recycling the acrylic fiber waste, it was found necessary to find an cost effective method for converting the acrylic fiber wastes in any form to acrylic powder which could be easily mixed with the virgin pulverized acrylic polymer and introduced in manufacturing process, minimizing the waste generation and reducing the production cost.

OBJECTS OF INVENTION:

The main object of the invention is to develop an economical and environment friendly waste utilization process for grey acrylic fiber waste.

Another object of this invention is to convert grey acrylic fiber waste generated during the fiber production into acrylic powder.

Yet another object of this invention is to devise a novel process whereby grey acrylic fiber waste could be converted into acrylic powder for its usage as a raw material for the acrylic fiber production line.

A further object of this invention is to develop a method to manufacture acrylic powder from acrylic fiber waste without affecting the original properties of virgin acrylic fiber.

An additional object of this invention is to propose a process whereby the solvents used during the process could be recycled to the system minimizing the effect to the environment and reducing the conversion cost.

An additional object of the invention is to minimize the use of water in precipitation process to reduce the chances of water pollution on account of additives in the fiber waste and solvents used in the process.

STATEMENT OF INVENTION:

According to this invention, there is provided a novel process for conversion of grey acrylic fiber waste to acrylic powder comprising of the steps of (a) dissolving the acrylic wastes in solvents such as N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), dimethylacetamide (DMAc), aqueous solution of sodium thiocyanate (NaSCN) but not limited to above mentioned solvents in the concentration of 1-25% and temperature of not more than 90°C; (b) filtration through any coarse filter media to remove any un-dissolved and other impurities before precipitation; (c) precipitation of acrylic polymer from the solution using any commercially available primary-, secondary- and tertiary alcohols in the ratio of 1:1 to 1:10 volume by volume of polymer solution and alcohols; (d) vacuum filtration to remove the solvents mixture; (e) dispersing the polymer in hot/cold DM water in the ratio of 1:5 to 1:20 weight by volume to remove residual solvents; (e)

palletizing the wet precipitate before drying or directly drying the polymer at elevated temperature to remove moisture; (f) pulverizing the dried polymer and sieving to obtain fine acrylic powder, (g) recovering the used solvents by distillation.

DETAILED DESCRIPTION OF THE INVENTION:

The present novel invention involves an economical and environment friendly process to convert grey acrylic fiber waste generated during the fiber production into acrylic powder which could be used as a raw material for the acrylic fiber production line without affecting the original properties of virgin acrylic fiber. In this process, solvents used can be recycled to the system minimizing the effect to the environment and reducing the conversion cost.

In the instant invention, grey acrylic fiber waste is dissolved in appropriate solvents such as N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), dimethylacetamide (DMAc), aqueous solution of sodium thiocyanate (NaSCN) but not limited to the given examples in the concentration of 1-25% weight by volume at a temperature not exceeding 95°C.

The polymer solution is then subjected to coarse filtration in order to remove non-acrylic foreign particles from the solution to get clear acrylic solution and the filtrate is mixed with any commercially available aliphatic primary, secondary and tertiary alcohols such as methanol, ethanol, n-propanol, n-butanol, isopropyl alcohol, tert- butyl alcohol or by combination of any of them in the ratio 1:1 to 1:10 of polymer solution : alcohol for effective precipitation process of dissolved acrylic polymer whereby

the solvent and precipitating agent/s are remove by filtration preferably vacuum filtration process.

The precipitates are suspended in adequate media such as alcohols or demineralised (DM) water in the concentration of 1:5 to 1:20 to remove the traces of DMF, DMAc, DMSO or NaSCN and repeated filtration process to obtain purified polymer.

The wet cake undergoes a drying process either in its existing form or by converting it to noodles using extruder before drying in continuous belt dryer followed by pulverization in which the acrylic polymer is converted to fine powdered before sieving and sending to storage bins.

The solvents used in the process are recovered and reused in subsequent process.

It is pertinent to mention here that the intrinsic viscosities of the recycled acrylic polymer powders were in the range of 1.40 ± 0.02 matching the intrinsic viscosity of the original acrylic polymers. Intrinsic viscosity is obtained by determining the relative viscosity for 0.2% W/V solution of polymer in 0.2 Molar Lithium Bromide/DMF Solvent at 25°C.

Examples

Example: 1

8 gm of acrylic fiber was dissolved in 100 ml of hot DMAc, cooled and precipitated with 180 ml of n-Propanol. The mixture is filtered and the white lumps were suspended in 100 ml of hot DM

water, filtered and procedure was repeated to ensure complete removal of DMAc and alcohol. Dried in oven and pulverized to have fine acrylic powder.

Example: 2

6.8 gm of acrylic fiber was dissolved in 100 ml of hot NaSCN aqueous solution (90% weight by volume), cooled and precipitated with 250 ml of Ethanol. The mixture is filtered and the white lumps were suspended in 100 ml of Ethanol. Filtered and again suspended in 200 ml of DM water to ensure complete removal of NaSCN traces and ethanol. Filtered and dried in oven followed by pulverization to get fine white acrylic powder.

Example: 3

7.5 gm of acrylic fiber was dissolved in 75 ml of hot DMSO, cooled and precipitated with 300 ml of n-Propanol. The mixture is filtered and the white lumps were suspended in 150 ml of hot DM water, filtered and procedure was repeated to ensure complete removal of DMSO and alcohol. Dried in oven and pulverized to have fine acrylic powder.

It is to be noted that the present invention is susceptible to modifications, adaptations and changes by those skilled in the art. Such variant embodiments employing the concepts and features of this invention are intended to be within the scope of the present invention, which is further set forth under the following claims:

WE CLAIM

1. A novel process for conversion of grey acrylic fiber waste to acrylic powder comprising of the steps of:

   a. dissolving the acrylic wastes in solvents such as N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), dimethylacetamide (DMAc), aqueous solution of sodium thiocyanate (NaSCN) but not limited to above mentioned solvents in the concentration of 1-25% and temperature of not more than 90°C;

   b. filtration through any coarse filter media to remove any un-dissolved and other impurities before precipitation;

   c. precipitation of acrylic polymer from the solution using any commercially available primary-, secondary- and tertiary alcohols in the ratio of 1:1 to 1:10 volume by volume of polymer solution and alcohols;

   d. vacuum filtration to remove the solvents mixture;

   e. dispersing the polymer in hot/cold DM water in the ratio of 1:5 to 1:20 weight by volume to remove residual solvents;

   f. palletizing the wet precipitate before drying or directly drying the polymer at elevated temperature to remove moisture;

   g. pulverizing the dried polymer and sieving to obtain fine acrylic powder;

   h. recovery of used solvents using distillation process.

2. A novel process as claimed in claim 1 where the concentration of acrylic fiber waste to solvent or solvents mixture is in the range of 1-25% weight by volume.

3. A novel process as claimed in claim 1 wherein the precipitating agent(s) is/are any commercially available aliphatic primary, secondary and tertiary alcohols.

4. A novel process as claimed in claim 3 wherein the precipitating agent(s) can be single or mixture of said aliphatic alcohols.

5. A novel process as claimed in proceeding claims wherein the purification of polymer precipitate is carried out hot/cold demineralised (DM) water in the concentration of 1:5 to 1:20.

6. A novel process as claimed in proceeding claims wherein the further dispersion and purification is done using hot/cold DM water to ensure complete removal of solvents from polymer precipitates.

7. A novel process as claimed in claim1 wherein the wet polymer is converted to noodles using extruder before drying in continuous belt dryer.

8. A novel process for conversion of grey acrylic fiber waste to acrylic powder as herein substantially described read in conjunction with the accompanying examples.

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. D01F13/04 C08J11/06 C08J3/16
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
D01F C08J B29B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 1 084 563 A (ZAKLADY WLOKIEN SZTUCZNYCH ANI) 27 September 1967 (1967-09-27) the whole document	1-8
A	DD 200 233 A1 (LOERZER ULRICH; PETER ANNELIESE; TRETNER WALTER; PETER EBERHARD; SCHMI) 30 March 1983 (1983-03-30) page 3 - page 4 examples	1-8
A	US 2 862 895 A (CUMMINGS JR PERCIVAL W ET AL) 2 December 1958 (1958-12-02) column 2, line 16 - column 3, line 47 examples	1-8

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

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