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METHOD FOR DYEING NYLON FILAMENTS

The invention relates to filaments for toothbrushes.

Breuer et al., U.S. Pat. No. 4,802,255
5 describes nylon monofilaments that include at least two
colored regions, one of which is provided by a dye.
The monofilaments are included in the bristles of
toothbrushes and, as the toothbrush is used by the
owner, the color intensity of the dyed region changes
10 in response to wear, providing an indication of when
the toothbrush should be replaced. Breuer et al. lists
six food dyes or colorants that can be used to dye the
monofilaments including Indigotine (FD&C Blue No. 2),
and in col. 4 broadly describes the conditions that can
15 be used for dyeing. Breuer et al. also provides a
number of specific examples of dyeing procedures,
including three (examples 4-6) in which Indigotine is
used as the dye.

The specific dyeing procedures described by
20 Breuer et al. were for laboratory samples. However, to
produce a sufficient amount of dyed filaments for
commercial toothbrushes, a procedure is needed that can
be used for dyeing large amounts of filaments. The
procedure should be cost-efficient and, of course,
25 should produce nylon filaments that have a consistent
level of dye penetration.

In general, the invention is a method for

dyeing batches of nylon filaments for toothbrushes. As used herein, a batch is at least two pounds of nylon filament. The method includes immersing a batch of a first nylon filament for between 10 minutes and 80 minutes in an aqueous solution containing between .01% and 0.1% weight to volume of Indigotine dye. The solution should have a pH between 1 and 7, and should have a temperature between 50°C. and 90°C. While the nylon filament is immersed, the dye penetrates into an outer cross-sectional region but not into an inner cross-sectional region. The batch of the first nylon filament is removed from the solution, and can be used for bristles in a wear-indicating toothbrush. The same procedure is repeated for a batch of a second nylon filament, reusing the same solution. Preferably, the same solution is then used for dyeing additional batches. In the preferred method, the aqueous solution contains between 0.01% and 0.05% weight to volume of Indigotine dye; the solution has a pH of between 2.5 and 4.5; and the temperature of the solution is between 70°C. and 85°C. Preferably, the dyeing step is performed at atmospheric pressure and takes less than 60 minutes, and the nylon filaments have a diameter of between 0.005 inches and 0.014 inches.

The method can be used to produce commercial quantities of nylon filaments for use in wear-indicating toothbrushes in a cost-effective manner. For example, the procedure can be used on 10-40 lb. batches of nylon filaments, and even on batches weighing 100 lbs or more. Importantly, the same solution can be reused at least once, and even up to five or six times, and still produce filaments in which the Indigotine dye has penetrated an even, consistent cross-sectional area. The method is easy to carry out, safe, and provides a stable dye solution.

Other advantages of the invention will be apparent from the description of the preferred embodiment thereof, and from the 'claims.

5 Dyed wear-indicating nylon monofilaments, toothbrushes including such monofilaments, and the way the toothbrushes are made are described in detail in Breuer et al.

10 The dyeing solution used in the method of the invention contains between 0.01% and 0.1% weight to volume Indigotine. If too much Indigotine is used, too much dye penetrates too far into the filament. If too little dye is used, too little dye may penetrate, and the solution may not be reusable because the initial batch of filament may absorb the limited quantity of dye present.

15 The temperature of the solution should be between 50°C. and 90°C. Too high a temperature results in water quickly evaporating off, resulting in the dye becoming more concentrated, and the dye being taken up too fast, making the degree of penetration more difficult to control. Too low a temperature results in too slow a dye uptake.

20 The dyeing process preferably is done at atmospheric pressure, to avoid the necessity of using large pressurized equipment in a plant.

25 Indigotine is an acid stable dye, and the solution should be acidic.

30 The volume of dyeing solution should be large enough so that the first batch of filament does not absorb such a high percentage of the dye in the solution that the solution cannot be reused to dye a second batch.

35 The nylon filaments, most preferably monofilaments, should be of the diameter commonly used in toothbrushes, most preferably 0.008 or 0.009 inch.

The nylon filament should be immersed in solution long enough to get adequate dye penetration of the outer cross-sectional diameter of the filament to provide good wear-indicating characteristics.

5 Preferably, the immersion time should be as limited as possible, and should be less than 1 hour.

10 A detailed description of the most preferred method is provided below, beginning with a description of the equipment and solutions used, followed by the procedure.

The dyeing step is performed in a 633 gallon Whiting stainless steel tank having side dimensions of 30" x 40" x 130".

15 The dyeing solution used in the tank includes 633 gallons of distilled water; 7.7 liters of glacial acidic acid; and 555 g of 100% pure Indigotine. The temperature of the solution is maintained at 75°C. $\pm$ 1.5°C.

20 Two 633 gallon rinse tanks also are used. The first tank is filled with tap water. The second tank is filled with tap water mixed with 1.5 liters of Dow Corning #24 silicone emulsion.

25 The monofilament used is an eight foot long, 0.008 inch diameter Wytex 6.12 S nylon hank (bundle) weighing approximately 10 pounds. The hank should be siliconed, annealed, wet, and combed prior to dyeing, according to standard procedures. The hank should not be dried prior to use.

Dyeing Procedure

- 30 1) Completely submerge four hanks in the dye solution.
- 2) Set timer for 45 minutes.
- 35 3) Lift and resubmerge hanks every 1.5 minutes to permit circulation of dye between filaments.

- 4) After 45 minutes, remove hanks and transfer to the first rinse tank. Rinse by dipping 4 to 5 times.
- 5) Transfer the to the second rinse tank. Continue rinsing by dipping another 4 to 5 times. This rinse will restore the finish lost during the dyeing process.
- 6) Recomb hanks and continue with the normal drying and wrapping process.
- 7) Repeat the procedure 4 times for additional batches of hanks, using the same solutions. The dye solution and first rinse are changed after 5 hanks have been dyed; the second rinse solution is replaced daily.

Optionally, the above procedure can be modified by immersing set of hanks for 30 minutes in a dyeing solution heated to $80^{\circ}\text{C.} \pm 1.5^{\circ}\text{C.}$ In addition, the procedure can be used to dye 10 hanks at a time, as opposed to four hanks.

Other embodiments are within the claims.



C L A I M S

1. A method of dyeing nylon filaments for toothbrushes, comprising:

5 (a) immersing a batch of a first nylon filament for between 10 minutes and 80 minutes in an aqueous solution comprising between 0.01% and 0.19% weight to volume of Indigotine dye, said solution having a pH between 1 and 7 and having a temperature between 50°C. and 90°C., said Indigotine dye
10 penetrating into said first nylon filament to color an outer cross-sectional region of said first nylon filament, and not penetrating into an inner cross-sectional region of said first nylon filament;

15 (b) removing the batch of said first nylon filament from said solution to provide a first nylon filament having said outer cross-sectional region colored with said Indigotine dye and said inner cross-sectional region not colored with said Indigotine dye; and

20 (c) repeating steps (a) and (b) for a batch of a second nylon filament, reusing the same said solution.

2. The method of claim 1, comprising the additional step of repeating steps (a) and (b) for a
25 batch of a third nylon filament reusing the same said solution.

3. The method of claim 2, comprising the additional step of repeating steps (a) and (b) for a batch of a fourth nylon monofilament, reusing the same
30 said solution.

4. The method of claim 1, wherein step (a) is performed at atmospheric pressure.

5. The method of claim 1, wherein said solution has a temperature of between 70°C. and 85°C.

35 6. The method of claim 1, wherein said solution



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comprises between 0.01% and 0.05% weight to volume of Indigotine dye.

7. The method of claim 1, wherein said solution has a pH of between 2.5 and 4.5.

5 8. The method of claim 1, wherein said first nylon filament and said second nylon filament have an average diameter of between 0.005 inches and 0.014 inches.

10 9. The method of claim 1, comprising the additional steps of affixing the first dyed nylon filament to the head of a toothbrush, and at least some of the second dyed nylon filament to the head of a toothbrush.

15 10. The method of claim 1, wherein said first nylon filament and said second nylon filament are monofilaments.

11. The method of claim 1, wherein said first nylon filament is immersed in said aqueous solution for less than 60 minutes.

20 12. The method of claim 1, wherein said batch of said first nylon filament weighs at least 10 lbs.

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METHOD FOR DYEING NYLON FILAMENTS

A B S T R A C T

A method for dyeing nylon filaments for use in wear-indicating toothbrushes includes immersing a
5 batch of a first nylon filament for between 10 minutes and 80 minutes in an aqueous solution containing between 0.01% and 0.19% weight to volume of Indigotine dye, said solution having a pH between 1 and 7 and having a temperature between 50°C. and 90°C., the dye
10 penetrating into the filament to color an outer cross-sectional region and not penetrating into an inner cross-sectional region of the filament; removing the batch of the first nylon filament from the solution, and repeating the steps for a batch of a second nylon
15 filament, reusing the same solution.

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REPLACEMENT SHEET

14 DEC 1998

CLAIMS

DATE OF RECEIPT 14/12/98

1. A method of dyeing nylon filaments for toothbrushes, comprising :

(a) wetting said nylon filaments and immersing a first batch of said nylon filament for between 10 minutes and 80 minutes in an aqueous solution comprising between 0.01% and 0.19% weight to volume of Indigotine dye, said solution having a pH between 1 and 7 and having a temperature between 50°C. and 90°C., said Indigotine dye consistently penetrating into said nylon filament to color an even outer cross-sectional region of said nylon filament, and not penetrating into an inner cross-sectional region of said nylon filament;

(b) removing the first batch of said nylon filament from said solution to provide a nylon filament having said outer cross-sectional region colored with said Indigotine dye and said inner cross-sectional region not colored with said Indigotine dye;

(c) repeating steps (a) and (b) for a second batch of said nylon filament, reusing the same said solution, said Indigotine dye consistently penetrating into said nylon filament to color evenly the same outer cross-sectional region of said nylon filament in said second batch as was colored in said first batch.

2. The method of claim 1, comprising the additional step of repeating steps (a) and (b) for a third batch of said nylon filament, reusing the same said solution, said Indigotine dye consistently penetrating into said nylon filament to color evenly the same outer cross-sectional region of said nylon filament in said third batch as was colored in said first batch.

3. The method of claim 2, comprising the additional step of

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repeating steps (a) and (b) for a fourth batch of said nylon filament, reusing the same said solution, said Indigotine dye consistently penetrating into said nylon filament to color evenly the same outer cross-sectional region of said nylon filament in said fourth batch as was colored in said first batch.

4. The method of claim 1, wherein step (a) is performed at atmospheric pressure.

5. The method of claim 1, wherein said solution has a temperature of between 70°C. and 85°C.

6. The method of claim 1, wherein said solution comprises between 0.01% and 0.05% weight to volume of Indigotine dye.

7. The method of claim 1, wherein said solution has a pH of between 2.5 and 4.5.

8. The method of claim 1, wherein said nylon filaments have an average diameter of between 0.127 and 0.356 mm (0.005 inches and 0.014 inches).

9. The method of claim 1, comprising the additional steps of affixing the first dyed nylon filament to the head of a toothbrush, and at least some of the second dyed nylon filament to the head of a toothbrush.

10. The method of claim 1, wherein said nylon filaments are monofilaments.

11. The method of claim 1, wherein said first batch of said nylon filament is immersed in said aqueous solution for less than 60 minutes.

12. The method of claim 1, wherein said first batch of said nylon filament weighs at least 4.8 kg (10 lbs.).