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3,236,825 POLYVINYL CHLORÍDE FIBRES AND PROCESS FOR PRODUCING SAME

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2 Claims. (Cl. 260--92.8)

The present invention relates to new and improved polyvinyl chloride fibres, which have only a small shrinkage in the organic solvents normally used for the dry cleaning of clothing, particularly trichlorethylene and perchlorethylene.

It is known to manufacture filaments, yarns, fibres, rovings and the like, which will hereinafter be included in this specification in the term "fibres," from vinyl chloride polymers, either by wet spinning or dry spinning 20 solutions of these polymers in the appropriate solvents.

The fibres obtained by the aforesaid processes are of value in the textile field, because they have chemical inertness, high resistance to light, non-flammability and strong thermal, electrical and acoustic insulation prop-25erties.

However, these fibres when given an oriented crystalline structure are thermoplastic and sensitive to solvents. Their thermoplasticity is shown by a shrinkage of the fibres under the influence of heat. This shrinkability has 30 found numerous applications for obtaining special effects in woven knitted and non-woven fabrics. However, it constitutes a disadvantage for other applications.

Processes for reducing this heat sensitivity have been investigated, either by employing polymers with special characteristics, or especially by subjecting fibres which have already undergone a stretching to heat setting treatments under tension.

Such fibres, even though set, are sensitive to certain 40 organic solvents used for the dry cleaning of clothing. The most usual of these solvents are: light petroleum spirit, white spirit, benzene, carbon tetrachloride, trichloroethylene and perchlorethylene. Among these solvents, trichlorethylene has a high swelling power and is the most aggressive, this swelling of the fibres being ac-45companied by considerable shrinkage. The attack is stronger as the temperature is higher.

In dry cleaning installations, the operations should theoretically be carried out in the cold, but in practice the working conditions are such that the temperature of the solvent is sometimes in the range of about 35 to 40° C. For this reason, a temperature of 40° C. has been chosen as safety limit.

The shrinkage in trichlorethylene of already known vinyl chloride polymer fibres can reach 40% and even 60% in certain cases. No process capable of avoiding this disadvantage has so far been found.

In the following description of the present invention, reference will be made solely to trichlorethylene, since this is the solvent which has the highest swelling power.

According to the present invention it is possible to obtain polyvinyl chloride fibres which are characterised by having a good breaking strength, an improved stability in boiling water and a shrinkage less than or equal to 65 10% after 30 minutes in trichlorethylene at 40° C. In particular, by the present invention it is readily possible to obtain polyvinyl chloride fibres having the entirely novel properties of a breaking strength of more than 2 g. per denier, a shrinkage in boiling water of less than 10% and a shrinkage in trichloroethylene at 40° C. of less than 10%.

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These fibres are obtained by spinning vinyl chloride polymers obtained in known manner by polymerisation at low temperature, i.e. below 0° C., the AFNOR index of these polymers being at least equal to or greater than 450 (index determined in cyclohexanone at 25° C. according to the French Standard: NF T 51,013). The AFNOR index equals

 $\frac{\frac{n}{n_o}-1}{C}$

where n equals the dynamic viscosity of the solution, n_o equals the dynamic viscosity of the pure solvent, and C equals the concentration in grams per milliliter of the 15 solution. The fibres can be spun by known methods, such as dry spinning or wet spinning of solutions or suspensions of polymers in any suitable solvent or mixture of solvents. The polymers may contain plasticisers, pigments, loading agents, dyes, stabilisers or other products capable of modifying certain of their properties, such as

for example their affinity for dyeing and their electrical resistivity. As is customary with the yarns of synthetic polymers,

the filaments obtained from these polymers are subjected to a drawing operation with a view to giving them an oriented crystalline structure. For the present polymers, this operation should preferably be carried out at high temperature and more especially at a temperature between 100 and 150° C.; they are then subjected to a

heat treatment under tension at a temperature which may reach 170° C. The drawn filaments, before or after the heat setting treatment, may be subjected to a heat relaxation treatment which reduces the sensitivity of the fibres to heat and to solvents. The extent of this relaxa-

35tion or shrinkage may vary according to the effect to be obtained; it could for example reach 30% if particularly stable fibres are desired. It is found that the filaments obtained are practically insensitive to the action of trichlorethylene.

The fibres obtained according to the invention may be subjected to appropriate finishing treatments to prepare them for weaving, knitting or the production of unwoven articles, and for these purposes they may be employed alone or mixed with other natural, artificial or synthetic fibres.

The following example illustrates the invention.

Example

Polyvinyl chloride fibres are obtained by spinning a 50 solution of a vinyl chloride polymer having the AFNOR index 510, which can be obtained by polymerisation at a temperature between -10 and -20° C. in the presence of suitable catalysts. The wet spinning process is used, starting with a 10% solution of the polymer in dimethyl formamide. The initial suspension in this solvent is 55brought to a temperature of 135° C., which causes the polymer to dissolve, the solution then being filtered and fed to a spinneret with 240 holes each having a diameter of 0.07 mm. The filaments are coagulated in a bath at 40° C. initially formed of 70% of dimethyl formamide 60 and 30% of water, the concentration being kept constant during the spinning. The filaments are then washed in baths with progressively smaller contents of dimethyl formamide. After drying, the fibres are drawn in mineral oil at 140° C. in the ratio of 1 to 12, then subjected to a heat treatment under tension at 150° C. for 30 seconds in mineral oil. The fibres obtained have the following properties:

Breaking strength _____g./den__ 4.2 Elongation at break _____percent__ 23 70Shrinkage in boiling water after 30 minutes __do____ 6.6

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It is found that the shrinkage at 40° C. in trichlorethylene is only 12% after 30 minutes.

If the drawn fibres are subjected to a relaxation treatment at 130° C. while permitting a shrinkage of 20% and are then heated under tension for 30 seconds in the mineral oil at 160° C., the new fibres obtained have the following properties:

Breaking strengthg./den 3	3.2	
Elongation at breakpercent	48	• •
Shrinkage in boiling water after 30 minutes do /	4.3	10
Shrinkage in trichlorethylene at 40° C. after 30		
minutesdo2	2.1	

In order to obtain this resistance to trichlorethylene, it is necessary for the AFNOR index of the polymer to 15 be high. Heat stabilised fibres obtained under similar conditions, starting with a vinyl chloride polymer obtained by polymerisation at a temperature below 0° C. but having an AFNOR index of only 135, although they may have good mechanical properties, nevertheless, show a 20 treatment to cause shrinkage of at most 30%. high shrinkage on immersion in trichlorethylene at 40° C. for 30 minutes of the order of 40 to 50%.

Hitherto, the polyvinyl chloride used for making fibres was obtained by emulsion polymerisation, suspension polymerisation or block polymerisation at temperatures 25 higher than 0° C. and had an AFNOR index between 100 and 200. The fibres showed shrinkages up to 60%in trichlorethylene at 40° C.

The drawing and the heat treatments applied to the 30 fibres of the present invention can be carried out in oil, air or in any appropriate non-swelling liquid medium or gaseous medium, or by contact with a hot surface.

The duration and the temperature of the heat treatment under tension of the fibres depends on properties which it 35 is desired to give to the fibres. After this heat treatment, a second heat relaxation treatment may be applied if desired.

Results similar to those obtained by wet spinning are also produced by dry spinning.

I claim:

1. Polyvinyl chloride fibres having a breaking strength of more than 2 g. per denier, a shrinkage in boiling water of less than 10% and a shrinkage in trichlorethylene at 40° C. of less than 10%.

2. Process for the production of polyvinyl chloride fibres having a breaking strength of more than 2 grams

per denier, a shrinkage in boiling water of less than 10% and a shrinkage in trichloroethylene at 40° C. of less than 10%, which comprises spinning a solution of polyvinyl chloride having an AFNOR index of 450 as determined in cyclohexanone at 25° C., where the AFNOR index equals

$$\frac{n}{n_{o}}-1}{C}$$

and *n* equals the dynamic viscosity of the solution n_0 equals the dynamic viscosity of the pure solvent C equals the concentration in grams per millilitre of the solution and obtained by polymerisation of vinyl chloride at a temperature below 0° C., stretching the resulting filaments at a temperature of 100 to 150° C., and thereafter subjecting them to two stages of treatment carried out in either order, one being heat setting at a temperature of at most 170° C. and the other being a heat relaxation

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