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TREATING AGENT FOR THERMOPLASTIC SYNTHETIC FIBERS

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ABSTRACT OF THE DISCLOSURE

A treating agent for thermoplastic synthetic fibers comprising

(1) 50–99.5% by weight of a specific ethylene oxide/alkylene oxide copolymer,
(2) 0.5–30% by weight of a specific polymethylphenyl siloxane, and
(3) 0–49.5% by weight of another oil component than components (1) and (2) above,

the amounts of the respective components being based on the total weight of the treating agent.

This invention relates to a novel treating agent for thermoplastic synthetic fibers.

Generally, the production of yarns from an organic linear synthetic polymer such as polyesters and polyamides requires the steps of forming undrawn yarns by a suitable spinning method such as melt-spinning, wet-spinning or dry-spinning method, and then hot drawing the undrawn yarns. Usually, for improvement of the properties, the yarns are then heat set. Furthermore, in the production of bulky yarns such as yarns of woolen finish, the yarns must be heat-treated to set their shapes. The various thermal treatments required in the production of thermoplastic synthetic fibers are performed while the yarns are running at a very high speed. In order to perform these thermal treatments without trouble, it is absolutely necessary to impart to the yarns both smoothness (the property of the yarn to make smooth contact with an object such as a guide or heater) and collectivity (the property of the individual fibers of the yarn to gather close together without separation) by treating the yarns with a suitable treating agent, as is usually done.

In recent years, increased speeds of production have been demanded, and with it, the speed of running yarns at the time of the above-mentioned thermal treatments has become higher. In order to subject the yarns fully to the thermal treatment, it is necessary to elevate the treating temperature. For better heat transfer, the use of a heated plate of the contact type has frequently been used as the heating means. The employment of such process conditions, however, results in the degeneration of the treating agent due to high temperatures, and a stronger tendency of the degenerated matter to stick to the plate. Furthermore, wastes of fibers are generated from the yarns to be treated, and stick to the heated plate. Thus, the wastes of the fibers similarly undergo heat degeneration, and there is a stronger tendency towards the formation of thermally degraded matters. The thermally degraded matters resulting from the oil and wastes of fibers deposit on the heated plate, which in turn causes a reduction in

the quality of the final product. Furthermore, in order to maintain the quality of the product, the heated plate should be cleansed with increasing frequency, and this finally leads to a decreased efficiency of production.

Examples of the treating agent which has previously been used include mineral oils as smoothening agents, or esters of acids and higher alcohols. These oils have the defect that when the treating temperature is raised, not only does fuming occur, but also the resultant tar-like material is accumulated on the plate and is very difficult to remove.

Accordingly, in order to produce products of good quality with good efficiency, there has been a great demand for treating agents which not only impart high degrees of smoothness and collectivity to yarns, but also do not cause the accumulation of the heat degraded products of the treating agent or wastes of fibers on the heated plate over which the yarns pass, which degraded products on the heated plate can be easily removed by cleansing. These treating agents, from the standpoint of cost and safety of operation, should be inexpensive and free from flammability and toxicity, and should be applied to the yarns in the form of an aqueous emulsion. Accordingly, whether the treating agent can be dispersed as a stable aqueous emulsion is one of the very important factors for determining the industrial suitability of the treating agent.

Generally, in order to prevent the accumulation of a tar-like material on a heated plate by the selection of the treating agent, it is necessary to use a treating agent which is stable without decomposition even during a long time stay on the heated plate and moves always in the liquid state together with the yarns, or one which is volatilized immediately upon deposition on the plate.

Those compounds which are a stable liquid at a treating temperature as high as 220° C. to 300° C. used in the high speed production of the yarns are the so-called silicones, that is, polydimethylsiloxane and its derivatives having a part of the methyl groups substituted with a phenyl group. It is difficult however to make an aqueous emulsion of these substances. If an alkyl group or ethylene oxide is added to the terminals of the polydimethyl siloxane in order to increase its emulsifiability, the resulting compound tends to be thermally degraded, and solidifies on a high temperature plate held above 220° C.

In order to remove this defect, a method has been proposed to apply polydimethyl siloxane directly to the yarns without making it into an aqueous emulsion, but this method has the defect that sufficient collectivity cannot be obtained.

An example of an organic treating agent which is relatively stable at a temperature in excess of 220° C. is polyphenylene oxide but this treating agent has the defect of readily undergoing the influences of the radical decomposition of other additives.

Therefore, the conventional smoothening agents undergo heat degeneration at high temperatures above 220° C. and volatilize, and at the same time, form tar-like substances which are difficult to remove. Those compounds which have resistance to heat degeneration have the defect of poor collectivity or difficulty in forming an aqueous emulsion thereof. Another conceivable method of forming a tar-like substance that involves the use of a very highly volatile oil and the provision of a smoke-absorbing device. However such a contrivance alone does not give sufficient smoothness.

Accordingly, an object of the present invention is to provide a novel treating agent for thermoplastic synthetic fibers which is free from the above-mentioned defects. The treating agent of the invention can be used in the form of an aqueous emulsion, give excellent smoothness and collectivity to the yarns, and does not cause the contamination of the plate during the heat-treatment process. Even if the plate is contaminated, the contaminant can be readily removed by cleansing.

According to the present invention, there is provided a treating agent for thermoplastic synthetic fibers is provided, such agent comprising

(1) 50–99.5% by weight of an ethylene oxide/alkylene oxide copolymer consisting of at least one member selected from the group consisting of block copolymers of ethylene oxide and other lower alkylene oxides, random copolymers of ethylene oxide and other lower alkylene oxides, and such copolymers whose terminals are blocked with a lower alkyl group, each of the copolymers having an average molecular weight of 300 to 20,000, such copolymer component containing a total of 20–95% by weight of ethylene oxide,

(2) 0.5 to 30% by weight of a polymethylphenyl siloxane consisting of at least one polymethylphenyl siloxane having a phenyl content of not less than 15 mol percent and a kinetic viscosity measured at 30° C. of 10 to 10,000 centistokes, the phenyl group of the siloxane being optionally substituted with a halogen atom selected from the group consisting of fluorine, chlorine and brome, and

(3) 0 to 49.5% by weight of another oil component different than components (1) and (2) above,

the amount of the respective components being based on the total weight of the treating agent.

The principal features of the present invention are that the ethylene oxide/alkylene oxidice copolymer as a first component and the polymethylphenyl siloxane as a second component are used in the specific ratios set forth in the present invention, whereby the merits of the respective components are retained, and the demerits of one component are made up for by the other component. As a result, a treating agent having superior properties which can not be obtained with the conventional treating agents can be provided by the synergistic effect of both components. Only when the specific limitations described in the present specification are met, are the first and the second components compatible with each other to form a stable aqueous emulsion, and such an emulsion can impart superior smoothness and collectivtiy to synthetic fibers. There is a very reduced tendency of forming a tar-like deposition on the heated plate, and such a deposit on the plate can be readily removed. If desired, a third component consisting of an ordinary compound may be used together with the first and second components. In order not to impair the superior properties brought about by the combination of the first and second components, it is necessary that the amount of the third component should be limited to not more than 49.5% by weight.

The ethylene oxide/alkylene oxide copolymer as a first component of the treating agent of the present invention (which may often be abbreviated to PAO–PEO in the present specification) is a block or random copolymer whose main chain is composed of ethylene oxide and another alkylene oxide such as propylene oxide or butylene oxide, or a copolymer whose terminals are blocked with a lower alkyl group. These are used either alone or in admixture. The entire first component should have an ethylene oxide content of 20 to 95% by weight, preferably 30 to 90% by weight. The ethylene oxide content, used herein, means a percent ratio of the total weight of the ethylene oxide unit contained in the first component to the total weight of the ethylene oxide unit and the unit derived from the other alkylene oxide contained in the first component. Furthermore, the respective ethylene oxide/alkylene oxide copolymer which makes up the first component should have an average molecular weight of 300 to 20,000, preferably 500 to 5,000. Block copolymers and random copolymers of ethylene oxide and other lower alkylene oxides, and such copolymers in which the terminals are blocked with a lower alkyl group are known per se, and can be produced by methods well known to those skilled in the art. In the present specification, a symbol such as PPG 1000 EO (20) is used to express the block copolymer. This symbol means that the block copolymer is one which is obtained by adding 20 mols of ethylene oxide (EO for short) to polypropylene glycol (PPG for short) having an average molecular weight of 1000 obtained by polymerization of propylene oxide. PPG 2000 EO (30), PPG 2000 EO (40), or PPG 3000 EO (10) is a block copolymer suitable for use in the present invention.

In the present specification, a symbol such as PO (30) EO (30) is used to express the random copolymer. This symbol means that it is a random copolymer of 30 mols of propylene oxide (PO for short) and 30 mols of ethylene oxide (EO for short). PO (40) EO (50) or BO (30) EO (40) (BO representing butylene oxide) is suitable for use in the present invention.

The blocking agent used for blocking the terminals of the random copolymer or block copolymer used in the present invention is a lower alkyl compound which contains active hydrogen capable of reacting with an epoxy group or can react with a terminal hydroxyl group, preferably those compounds having 1 to 6 carbon atoms. Examples of the lower alkyl compound include alcohols such as methanol, ethanol, propanol, hexanol ethylene glycol or glycerine, alkylamines such as ethylamine, propylamine, hexylamine or ethylene diamine, and organic acids such as acetic acid, propionic acid, or caproic acid. The blocking agent may be added together with AO and EO in the polymerization stage, or can be added after forming a block or random copolymer. For example, there are end blocked products of block copolymers, such as butoxy PPG 2000 EO(40) obtained by adding PO to butanol, and further adding EO to butoxy PPG 2000, or α-butoxy, ω-acetyl (PPG 2000 EO (40)) obtained by acetylating the remaining hydroxyl group on one terminal with acetic acid; and also end-blocked products of random copolymers such as butoxy (PO (30) EO (30)) obtained by adding 30 mols of PO and 30 mols of EO at random to one mol of butnol, or α-butoxy, α-caproyl (PO (30) EO (30)) obtained by esterifying such random copolymer with caproic acid.

The content of the first component in the entire treating agent should be 50 to 99.5% by weight.

If the molecular weight of the ethylene oxide/alkylene oxide copolymer which constitutes the first component is less than 300, sufficient collectivity cannot be imparted to fibers. If the molecular weight exceeds 2000, the viscosity becomes so high that it is not possible to handle the treating agent with ease. If the ethylene oxide content is less than 20% by weight, the dispersibility of the treating agent in water is reduced, and a good aqueous emulsion cannot be formed at room temperature. On the other hand, if this content exceeds 95% by weight, the volatility of the treating agent decreases, and it does not efficiently work as a smoothening and emulsifying agent. If the blend ratio of the first component in the treating agent of the present invention is less than 50% by weight, there is an increasing contamination of the plate, and if this ratio exceeds 99.5% by weight, it is difficult to remove the deposit on the plate.

The second component of the treating agent of the present invention is polymethylphenyl siloxane obtained by substituting some of the methyl groups of polydimethyl siloxane by a phenyl group or a phenyl group substituted with a halogen atom selected from fluorine, chlorine and bromine. The second component has a phenyl content of more than 15 mol percent especially 20 to 50 mol percent, and a kinetic viscosity of 10 to 10,000 centi-

stokes. The polymethyl phenyl siloxanes are used alone or in admixture. The phenyl content, as used herein, represents a proportion in mole percent of side phenyl groups or side substituted-phenyl groups to the total side chains of the polysiloxane. The kinetic viscosity is measured in a constant temperature tank at 30° C. by a customary method using a Cannon-Fenske type viscometer.

The compatibility of the polymethylphenyl siloxane with the first component depends upon the phenyl content and kinetic viscosity of the siloxane. Generally, the compatibility increases with increasing phenyl content and decreasing kinetic viscosity.

If the phenyl content of the polymethylphenyl siloxane is less than 15 mol percent, it cannot be emulsified even with the aid of the emulsifying action of the first component. There is no particular upper limit to the phenyl content, but actually, it is difficult to produce a second component with a phenyl content of above 50 mol percent. If the kinetic viscosity of the second component is less than 10 centistokes, the volatility of the treating agent becomes too high to give effects. On the other hand, if the kinetic viscosity exceeds 10,000 centistokes, it becomes difficult to handle the treating agent because of high viscosity. If the proportion of the second component is less than 0.5% by weight, the soil on the plate is difficult to remove. If it exceeds 30% by weight, the emulsifiability of the second component exceedingly decreases even with the action of the first component. An attempt to increase the emulsifiability by addition of the third component results in an increase contamination of the heated plate.

If desired, not more than 49.5% by weight, based on the total weight of the treating agent, of a third component may be added in order to modify the properties of the treating agent. The third component is an optional component, and a treating agent which does not contain the third component at all is also included within the scope of the present invention. The third component used in the present invention includes various oils which have previously been used as textile treating agents. Lubricant, emulsifying, anti-static, emulsion stabilizing, and oiliness improving agents are especially well known. Of these known oil components, preferred are those which do not substantially form a tar-like substance upon heating. Some examples of these oils are as follows. Examples of the lubricant includes esters of aromatic carboxylic acids and monohydric alcohols, such as trilauryl trimellitate or dioleyl phthalate, esters of monohydric alcohols and fatty acids, such as dioctyl sebacate, tridecyl isostearate, oleyl oleate, or oleyl laurate, esters of polyhydric alcohols and monobasic fatty acids, for instance, pentaerythritol tetraoctanate, trimethylol propane tridecanate, or diethylene glycol dioleate, and paraffinic compounds such as 450 second mineral oil or 100 second mineral oil. Those having no unsaturated group and a relatively low molecular weight, such as butyl stearate or 45 second mineral oil are preferred. Such lubricant can be used in relatively high proportions.

Generally, non-ionic surface active agents are used as the emulsifiers, which are long-chain alkyl compounds with active hydrogen having ethylene oxide added thereto. Examples of the long-chain alkyl compound having active hydrogen include high fatty acids such as oleic acid, stearic acid, or lauric acid, higher alcohols such as half esters of fatty acids with trimethylolpropane, sorbitan, oleyl alcohol, stearyl alcohol, methyl alcohol, lauryl alcohol, or octyl alcohol, and higher alkyl amines such as stearylamine, or laurylamine, and alkylphenols such as dodecylphenol or nonylphenol. Of these emulsifying agents, relatively easily volatile emulsifiers such as lauryl alcohol-ethylene oxide adduct, or laurylamine-ethylene oxide adduct, which have no unsaturated groups on the oleophilic groups are preferred in order to raise the proportion of the emulsifying agent in the treating agent of the present invention.

Various ionic surface active agents are used as the antistatic agents, the examples including amphoteric surface active agents of the imidazoline or betaine type, cationic surface active agents such as stearylammonium sulfate, or dimethylstearyl amine nitrate, and anionic surface active agents, for example, sulfates such as sulfated castor oil, sulfonates such as laurylsulfonate Na salt, or phosphates such as laurylphosphate K salt. Generally, many of the ionic surface active agents are non-volatile, and desirably the proportion of the antistatic agent should be minimized. Of these, especially preferred are phosphorus-containing surface active agents having good thermal stability. Isocetyl phosphate K salt which is oil soluble is a preferred example of the additive. Other examples of the emulsifier are higher fatty acids and higher alcohols. Agents which are used for improving oiliness include, for example, amine salts of fatty acids, and diketones.

Generally speaking, relatively easily volatile compounds free from unsaturated alkyl groups are preferably chosen as the third component of the treating agent of the present invention. From this point of view, polyalkylene oxide derivatives which do not come within the first component of the treating agent of the invention are most preferably used.

Almost all thermoplastic synthetic fibers can be treated by the treating agent of the invention, the examples including fibers made of polyesters, polyamides, polyacrylonitrile, polyolefins, or polyvinyl chloride, and there is no particular limitation. The treating agent of the present invention exhibits very superior treating effects at high temperatures at which the conventional treating agents are not usable. The treating agent of the present invention is especially applicable to such thermoplastic synthetic fibers as polyesters, for example, polyethylene terephthalate or polyethylene-2,6-naphthalate, and polyamides, for example, polyphenylene isophthalamide.

The treatment of the thermoplastic synthetic fibers with the treating agent of the invention can be effected by any method practiced for thermal treatment. Usually, the treating agent of the invention is applied to fibers as an aqueous emulsion having a concentration of 2 to 30% by weight. Sufficient treatment effects are obtained with a pick-up of the treating agent of usually 0.05 to 2.0% by weight based on the weight of the fibers. The temperature at which the fibers are heat-treated usually ranges from 150° C. to 320° C. The treating agent of the present invention exhibits especially good characteristics in the temperature range of 230–320° C.

The present invention will be further described by the following examples, in which the figures showing the compositions of treating agents are expressed in parts by weight.

EXAMPLE 1

Each of the treating agents of various compositions shown in Table 1 was heated to 50 to 80° C. Ten kilograms of the treating agent were taken, and poured into 90 kg. of water with stirring to form a 10% aqueous emulsion of the treating agent. Those emulsions which separated into layers within one day were not used in subsequent steps.

Polyethylene terephthalate having an intrinsic viscosity (η), as measured in o-chlorophenol at 30° C., of 0.65 was melted, and spun at a spinning temperature of 290° C. from a spinneret having circular spinning nozzles with 24 holes and a diameter of 0.30 mm. The emulsion prepared above was adhered to rollers, and the yarn was wound up at a rate of 1500 meters per minute. The speed of the feed rollers was adjusted so that the pick-up of the treating agent was 0.6% by weight. The resulting undrawn yarn was drawn to 3.04 times the original length between heated rollers at 83° C. and draw rollers. The drawn yarn was false-twisted at a rate of 1000 meters per minute using a high speed false twister directly con-

nected to the drawing device (the false twister has a total length of 2 meters, and is adapted to give false-twisting to a yarn by heat-setting it by a contact-type plate held at 250° C. and false-twisting it by the friction of two rotating discs). The characteristics of the treating agents were determined by the following standards.

(1) Emulsifiability

⊚ ---------- Semi-transparent to transparent emulsion, and stable for more than a week.
○ ---------- Semi-transparent emulsion, and stable for 3 days to one week.
Δ ---------- Semi-transparent to milk white, and stable for 1 to 3 days.
x ---------- Milk white, and separation occurs within one day.

(2) Contamination of the plate

⊚ ---------- Hardly any deposit on the plate after operating for two weeks.
○ ---------- A small amount of deposit is seen on the plate after operating for 1 to 2 weeks.
Δ ---------- A deposit resulting from thermal degradation of the treating agent is seen all over the plate. After operating for one week, and further operation impossible.
x ---------- A deposit resulting from thermal degradation of the treating agent is seen all over the plate after operating for three days, and further operation impossible.

(3) Ease of removing the contamination

⊚ ---------- Removal effected by a light rubbing with a metal brush.
○ ---------- Difficult to remove with a metal brush, but removal possible with the rubbing of a metal spatula.
Δ ---------- Removal effected by a strong rubbing with a metal spatula.
x ---------- Difficult to remove even by a strong rubbing with a metal spatula.

(4) Fuzzes of processed yarns

The resulting processed yarns were warped with a warper, and the number of fuzzes generated at this time per 10^6 m. was measured.

	Fuzzles per 10^6 m.
⊚	4 or less.
○	5 to 10.
Δ	11 to 30.
x	31 or more.

TABLE 1

	Runs numbers																			
	Invention				Comparison						Invention				Comparison					
Components	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Organopolysiloxane:																				
Polymethylphenyl siloxane phenyl content 45 mol percent, kinetic viscosity 450 c.s.	8										0.5				35					
Polymethylphenyl siloxane phenyl content 25 mol percent, kinetic viscosity 100 c.s.	8										5									
Polymethylphenyl siloxane phenyl content 20 mol percent, kinetic viscosity 20 c.s.	8										30	10			0.1		10	8	8	
Polymethylphenyl siloxane phenyl content 10 mol percent, kinetic viscosity 100 c.s.	8																			
Polydimethyl siloxane phenyl content 0 mol percent, kinetic viscosity 100 c.s.	8																			
Polymethyl chlorophenyl siloxane phenyl content 30 mol percent, kinetic viscosity 200 c.s.	8																			
Stearyl-terminated polydimethyl siloxane (30 c.s.)	8																			
Polyethylene oxide-terminated polydimethyl siloxane (70 c.s.)	8																			
Polymethyl(polyethylene oxide) siloxane (100 c.s.)	8																			
Polymethyl dodecyl siloxane (100 c.s.)	8																			
Alkylene oxide ethylene oxide copolymer:																				
Polypropylene glycol/ethylene oxide adduct molecular weight 250, ethylene oxide content 40 wt. percent																				90
Polypropylene glycol/ethylene oxide adduct molecular weight 522, ethylene oxide content 42 wt. percent					20					10	10			20	30					
Polypropylene glycol/ethylene oxide adduct molecular weight 3,330, ethylene oxide content 40 wt. percent	30	30	30	30	30	30	30	30	30	30	20	40		10	10	30		20		
Polypropylene glycol/ethylene oxide adduct molecular weight 3,440, ethylene oxide content 13 wt. percent								10					10	10						90
Butoxypolypropylene glycol/ethylene oxide adduct molecular weight 460, ethylene oxide content 50 wt. percent	20					20			20					20						
Butoxypolypropylene glycol/ethylene oxide adduct molecular weight 2,000, ethylene oxide content 50 wt. percent			20	20	20		10									20				
Butanol-propylene oxide ethylene oxide random adduct molecular weight 1,700, ethylene oxide content 50 wt. percent	60	40	60	40	40		35	60			10	20			9.9		20			
Butanol-propylene oxide ethylene oxide random adduct molecular weight 5,000, ethylene oxide content 50 wt. percent						10		20			30		25		50					
Butanol-propylene oxide ethylene oxide random adduct molecular weight 2,200, ethylene oxide content 25 wt. percent					10	10					4.5									
Butylene oxide ethylene oxide random adduct molecular weight 2,800, ethylene oxide content 50 wt. percent											10									
α-butoxy, ω-acetyl (polypropylene glycol/ethylene oxide adduct molecular weight 2,100, ethylene oxide (content 45 wt. percent							10						10							
α-butoxy, ω-acetyl (propylene oxide/ethylene oxide random adduct) molecular weight 1,100, ethylene oxide content 50 wt. percent											25		20							
Other components:																				
Trimethylolpropane tridecanate																				30
100 second mineral oil																	30			30
45 second mineral oil													10	20						
α-butoxy, ω-acetyl polypropylene glycol with a molecular weight of 500					10								5							
Lauryl alcohol ethylene oxide adduct	2	2	2	2	2	2	2	2	2	2	5	5	10		5		5	2	2	10
Nonylphenyl ethylene oxide adduct						5		5							10					10
Oleic acid/ethylene oxide adduct					5	5											10			10
Alkylsulfonate Na salt																	2.5			5
Isocetyl phosphate K salt										5		5					2.5			5

TABLE 2

	Run Nos.	Characteristics: Emulsifiability	Contamination of plate	Ease of removing contamination	Fuzzes of processed yarns
Invention	1	◎	◎	◎	◎
	2	◎	◎	◎	◎
	3	◎	◎	◎	◎
	4	◎	◎	◎	◎
Comparison	5	×	—	—	—
	6	×	—	—	—
	7	0—◎	Δ	0	Δ
	8	0—◎	Δ	0	×
	9	0—◎	Δ	◎	×
	10	0	Δ	0	Δ
Invention	11	◎	◎	0	0
	12	0—◎	0	◎	◎
	13	◎	◎	◎	◎
	14	◎	0	◎	◎
Comparison	15	◎	◎	Δ	×
	16	Δ	×—Δ	◎	Δ
	17	0	×	0	—
	18	0	◎	◎	×
	19	×	—	—	—
	20	◎	×	×	—

It is seen from the results shown in Tables 1 and 2 that the treating agents of the present invention (Runs Nos. 1 to 4 and 11 to 14) have good emulsifiability and can be used as aqueous emulsions, and cause little contamination of the plate, and a small amount of contamination can be readily removed, and that even when the yarn contacts a plate held at high temperatures, high degrees of smoothness and collectivity can be maintained, and therefore, the fuzzes of the processes yarns occur very little. On the other hand, the treating agents outside the scope of the present invention have some defects or other. Run No. 20 concerns the use of a conventional treating agent, which was found to cause considerable contamination of the plate, and consequent failure of operation in 1 to 2 days. Since the operating time is so short, the fuzzes of the processed yarn could not be determined. Run No. 5 relates to the use of polymethylphenyl siloxane having a phenyl content of less than 15 mol percent, and Run No. 6, to the use of polydimethylsiloxane with no phenyl content. In both cases, an aqueous emulsion could not be formed even with the aid of the PAO/PEO copolymer as the first component of the treating agent of the invention. Runs Nos. 7 to 10 relate to the use of other polysiloxanes obtained by introducing alkyl or polyethylene oxide in polydimethyl siloxane, instead of the second component of the treating agent of the invention. These polysiloxanes could be emulsified with the aid of the PAO/PEO copolymer as the first component, but causes increased contamination of the plate and increased number of fuzzes of the processed yarn. Run No. 15 illustrates the case where the amount of polymethylphenyl siloxane is less than 0.5% by weight. In this run, the contamination of the plate is difficult to remove, and there is an increasing number of fuzzes. Run No. 16 demonstrates that if the amount of the polymethylphenyl siloxane exceeds 30% by weight, not only does the emulsifiability of the treating agent decrease, but also the contamination of the plate increases. Run No. 17 demonstrates that when the amount of the PAO/PEO copolymer is less than 50% by weight and the emulsifiability is increased by addition of a third component, there is a considerable increase in the contamination of the plate, and the fuzzes of the processed yarn could not be determined. Runs Nos. 18 and 19 relates to the use of PAO/PEO copolymers which have different molecular weight and ethylene oxide content respectively from those specified in the invention. In Run No. 18, the yarn lacked both smoothness and collectivity, and in Run No. 19, the emulsifiability of the treating agent is very poor at room temperature.

EXAMPLE 2

Each of the treating agents of the compositions indicated in Table 3 was heated to 50° C. Eight kilograms of the heated treating agent were taken, and poured into 92 kg. of water held at 20 to 40° C. to form an 8% aqueous emulsion of the treating agent. Those emulsions which separated into layers within one day were not used in subsequent steps.

Polyethylene terephthalate was melt-spun under the same conditions as set forth in Example 1, and each of the resulting emulsions was applied to undrawn yarns. The yarns were drawn, and false-twisted in the same way as set forth in Example 1 except that the temperature of the contact-type plate was changed to 240° C., and the speed of false-twisting was changed to 800 meters per minute.

The operation was stopped after a lapse of one week. The emulsifiability, the contamination of the hot plate, and the number of fuzzes of the processed yarns were measured to show the characteristics of the treating agents of the present invention. The drawability of the undrawn yarns was determined according to the state of the fibers on the draw rollers, and the breakage of the yarns. Good drawability represents those undrawn yarns which are completely free from break with little occurrence of fuzzes, laps and the like on the draw rollers. The results are shown in Table 4.

TABLE 3

	Invention			Comparison				
	Run numbers							
Components	1	2	3	4	5	6	7	8
Alkylene oxide/ethylene oxide copolymer:								
PPG 2,000 EO (45)	70				100		80	
Butoxy (PO (30) EO (30))		80						
PO (20) EO (30)			90					
PPG 175 EO (2)*				10				75
Organopolysiloxane:								
Polymethylphenyl siloxane phenyl content 20 mol percent, kinetic viscosity 20 c.s	30		5					20
Polymethylphenyl siloxane phenyl content 35 mol percent, kinetic viscosity 80 c.s		10				100		
Polymethylphenyl siloxane phenyl content 10 mol percent, kinetic viscosity 150 c.s							20	
Other component:								
Trimethylolpropane tridecanate			30					
100 second mineral oil			30					
Oleyl alcohol/ethylene oxide adduct				20				
Lauryl alcohol/ethylene oxide adduct		5	20					5

*This copolymer has a molecular weight of 263, and cannot be the first component of the invention.

TABLE 4

	Invention			Comparison				
	Run numbers							
Characteristics	1	2	3	4	5	6	7	8
Emulsifiability	◎	◎	◎	◎	◎	×	×	Δ
Drawability	(1)	(1)	(1)		(1)	(1)	—	— [2]
Contamination of the plate	◎	◎	◎	×	Δ	—	—	◎
Fuzzes of the processed yarns	◎	◎	◎	—	×	—	—	×

[1] Good.
[2] Somewhat poor.

The above results demonstrate that the use of the treating agent of the present invention obviates the contamination of the hot plate and gives products of good quality with good efficiency, but that the conventional treating agents shown in Runs Nos. 4 to 8 have some defects or other. For example, in Run No. 4, the contamination of the plate was remarkable, and the operation became impossible in 3 days. Run No. 5 shows that the PAO/PEO copolymer alone causes lack of smoothness of the yarn on the hot plate, and there is an increasing number of fuzzes on the processed yarn. In Runs Nos. 6 and 7, the formation of emulsions was impossible, and therefore, the examination after drawing was not conducted. In Run No. 8, the collectivity of the yarns at the time of drawing is somewhat insufficient, and there is an increas-

ing number of fuzzes in the drawn yarns. Fuzzes also occurred considerably in the processed yarns.

EXAMPLE 3

Each of the treating agents of the compositions shown in Table 5 was heated to 50° C. Eight kilograms of the treating agent heated were taken, and poured into 92 kg. of water held at 20 to 40° C. to form an 8% aqueous emulsion of the treating agent. Those emulsions which separated into layers within one day were not used in subsequent steps. Using each of the resulting emulsions, polyethylene terephthalate was melt-spun, drawn and false-twisted. After operating for 2 weeks, the operation was stopped, and the contamination of the hot plate, and the ease of removing the contamination were measured in the same way as set forth in Example 1. The results are shown in Table 5 together with the emulsifiability of the treating agent.

TABLE 5

Component	Comparison	Invention						Comparison		
	Run numbers									
	1	2	3	4	5	6	7	8	9	10	11
Alkylene oxide/ethylene oxide copolymer:											
PPG 2,000 EO (35)	10	40	50	80	30	40	50	100	—	80	—
Butoxy (PO (30) EO (30))					60	50	45				
PPG 173 EO (2)						8					
Organopolysiloxane:											
Polymethyphenyl siloxane phenyl content 25 mol percent, kinetic viscosity 100 c.s.			30	—	8				50	—	100
Polymethylphenyl siloxane phenyl content 45 mol percent, kinetic viscosity 450 c.s.	35	—	15	—	2	1					
Polymethylphenyl siloxane phenyl content 10 mol percent, kinetic viscosity 100 c.s.										15	—
Other component:											
100 second mineral oil	50	25	10								
Lauryl alcohol/ethylene oxide adduct	40	5	10	5	2	—	4	—	20	5	—
Nonyl phenol/ethylene oxide adduct									30		
Emulsifiability	◎	△	◎	◎	◎	◎	◎	◎	△	×	×
Contamination of the plate	×	×	○	◎	◎	◎	◎	◎	×	—	—
Ease of removing contamination	×	◎	◎	◎	◎	◎	◎	×	◎	—	—

The results of these experiments show that the treating agents of the present invention (Runs Nos. 3 to 7) have good emulsifiability and can be used as aqueous emulsions, and that they cause little contamination of the hot plate, and the contaminant is easy to remove.

On the other hand, the treating agents used in Runs Nos. 1, 2 and 8–11 have some defects or other. In Run No. 1 the alkylenethylene oxide was emulsified well with a lubricant consisting predominantly of a mineral oil, but it caused remarkable contamination and failure of operation within one day.

In Run No. 2 in which the amount of the alkyl ethylene oxide is smaller than the range specified in the present invention, and the amount of the polymethylphenyl siloxane is large, the emulsifiability of the treating agent is reduced slightly, and there is considerable contamination of the hot plate although it can be removed easily.

On the other hand, in Run No. 8 in which the amount of the alkylene oxide is 100%, the removal of contamination is bad, and in Run No. 11 in which the amount of the polymethylphenyl siloxane is 100%, the treating agent is not emulsifiable, and is thus useless.

Furthermore, when the polymethylphenyl siloxane of the invention was emulsified without the aid of the PAO/PEO copolymer (Run No. 9), the contamination is easily removable, but the amount of the contamination is very large. When a polymethylphenyl siloxane having a phenyl content of less than 15 mol percent is used (Run No. 10), the treating agent cannot be emulsified, and is useless.

A wholly aromatic polyamide having an intrinsic viscosity [η], measured in 98% sulfuric acid at 30° C., of 1.9 and prepared from metaphenylene diamine and isophthaloyl chloride was dissolved in N-methyl pyrrolidone. The resulting spinning solution was wet-spun into an aqueous coagulating bath containing 40% of calcium chloride and 38% of N-methylpyrrolidone using a spinneret having 40 holes with a diameter of 0.12 mm. The spun filaments were taken up at a rate of 7 meters per minute, and continuously passed through a water-washing bath. The residual solvent was removed. The undrawn yarn was drawn to 3.15 times the original length in hot water at 90° C., and dried. A 10% aqueous emulsion of the treating agent of the invention having the composition of Run No. 1 in Example 1 or the comparative treating agent having the composition of Run No. 20 of Example 1 was applied to the drawn yarn so that the pick up of the treating agent reached 0.60% by weight. The yarn was then passed on a hot plate having a length of 50 cm. and a surface temperature of 310° C., and further drawn to 1.25 times on this hot plate to thereby obtain a 75 de./40 fil. drawn yarn. It was seen that in the case of using the treating agent of the invention, good drawn yarns were obtained for more than one week without cleansing of the plate, but that with the comparative treating agent, the contamination of the plate is remarkable, and without cleansing of the plate, good drawn yarns cannot be obtained after operating for one day.

EXAMPLE 5

Polyethylene-2,6-naphthalate having an intrinsic viscosity [η], measured in 6:4 phenol/o-dichlorobenzene at 35° C., of 0.65 was melt-spun at a spinning temperature of 320° C. using a spinneret having 24 circular holes with a diameter of 0.46 mm., and treated with the treating agent of the invention having the composition shown in Run No. 1 of Example 1 or the comparative treating agent having the composition of Run No. 20 of Example 1 as a 10% aqueous emulsion by the ordinary roller method. The yarn was wound up at a rate of 800 meters per minute. At this time, the speed of the rollers was controlled so that the pick-up of the treating agent was 0.60% by weight. The undrawn yarn obtained was drawn to 4.0 times its original length between heated rollers at 130° C. and draw rollers while being in contact with a plate held at 250° C., and wound up at a rate of 800 meters per minute to obtain a 75 de./24 fil. drawn yarn.

It was confirmed that with the treating agent of the present invention, there was hardly any contamination of the plate during 2 weeks operation, and the yarns could be drawn in good condition, whereas with the comparative treating agent, the contamination of the plate was considerable, and without cleansing the plate, good drawing of the yarn could not be performed after two days.

What we claim is:

1. A treating agent for thermoplastic synthetic fibers, said agent comprising

(1) 50–99.5% by weight of an ethylene oxide/alkylene oxide copolymer component consisting of at least one

member selected from the group consisting of block copolymers of ethylene oxide and another lower alkylene oxide selected from propylene oxide and butylene oxide, random copolymers of ethylene oxide and another lower alkylene oxide selected from propylene oxide and butylene oxide, and such copolymers whose terminals are blocked with a lower alkyl group of 1–6 carbon atoms, each of the copolymers having an average molecular weight of 300 to 20,000, said copolymer component containing a total of 20–95% by weight of ethylene oxide,

(2) 0.5–30% by weight of a polymethylphenyl siloxane component consisting of at least one polymethylphenyl siloxane having a phenyl content of not less than 15 mol percent and a kinetic viscosity measured at 30° C. of 10 to 10,000 centistokes, the phenyl group of the siloxane being optionally substituted with a halogen atom selected from the group consisting of fluorine, chlorine and bromine, and

(3) 0–49.5% by weight of another oil component other than components (1) and (2) above selected from the group consisting of lubricants, emulsifiers, antistatic agents, emulsification stabilizers, and oiliness improving agents for the thermoplastic synthetic fibers,

the amounts of the respective component being based on the total weight of the treating agent.

2. The treating agent of claim 1, wherein said thermoplastic synthetic fibers are selected from polyester fibers, polyamide fibers, polyacrylic fibers, polyolefin fibers, and polyvinyl chloride fibers.

3. The treating agent of claim 1, wherein said oil component (3) is selected from

esters of aromatic carboxylic acids and monohydric alcohols,

esters of monohydric alcohols and fatty acids,

esters of polyhydric alcohols and monobasic fatty acids,

paraffinic mineral oils,

alkylene oxide copolymers not included within scope of (1),

non-ionic surface active agents comprising long chain alkyl compounds with active hydrogen selected from high fatty acids, higher alcohols, higher alkyl amines and alkyl phenols, having ethylene oxide added thereto,

higher fatty acids,

higher alcohols,

amphoteric surface active agents of the imidozoline or betaine type,

cationic surface active agents,

anionic surface active agents,

amine salts of fatty acids, and

deketones.

4. The treating agent of claim 1, wherein each of the copolymers has an average molecular weight of 500–5,000.

5. The treating agent of claim 1, wherein said polymethylphenyl siloxane has a phenyl content of 20–50 mol percent.

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