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54 **Coating method.**

57 A method of coating using a slide hopper type coating method, an extrusion type coating method or a curtain hopper type coating die, in which (1) the coating solution is a mixture of two or more kinds of solvents having different boiling points and surface tensions, and the surface tensions are not more than 40 dyne/cm, and (2) the mixture of the solvents contains at least one surface orientating compound comprising an alkyl group, aralkyl group, aryl group or alkyl aryl group of which at least one hydrogen atom is substituted by a fluorine atom and the number of a carbon atom is 1 to 30, and (3) the added amount of the surface orientating compound is reduced from the amount of the surface tension of the solvent mixture by at least 0.5 dyne/cm, is disclosed. The coating method could provide obtaining uniform coatibility being free from coating defect, such as comet defect, tearing of composition defect etc..

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Field of the invention

The present invention relates to the coating method of the solvent-based coating solution, and specifically, the coating method by the bead coater or the curtain coater.

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Background of the invention

The slide hopper type or the extrusion hopper type coater are used widely as the coating equipment for photographic material and magnetic recording material because of the capability of high-speed, thin film and simultaneous multi-layer coating. This type of coater has a fountain for the coating solution called bead between the tip of coater, also referred to as edge or lip for short, and the running flexible substrate, or referred to as web, and solution is coated through this bead.

Such bead coater is largely influenced by the stability of the bead in order to stabilize coating. The stability of the bead is largely influenced by setting the gap between the lip and the web, environmental condition, physical property of coating solution, e.g., viscosity, surface tension, and especially for the simultaneous multilayer coating, the relationship of physical properties between layers. Furthermore, the stabilization of the bead becomes difficult when a coating speed increases or layer thickness becomes thin. When the bead is unstable, coating defects such as a streak defect, a comet defect, and a solution shortages will occur.

To improve such bead stability, the method of providing the pressure difference between the upper and lower side of the bead is known. Specifically, the bead is pulled downward by providing a vacuum room under the bead to enhance its stability.

However, even those methods sometimes cannot prevent comet defects, dropping defects and more streak defects caused by the coating solution oozing out of the bead on the lips other than bead and falling down on the film or support, when the coating solution having very low viscosity, such as the solution with low surface tension which contains organic solvent of not more than 10cps is used. It occurs even if coating speed is not too fast, and coating layer thickness is not too thin.

The object of the present invention is to provide the coating method capable of obtaining the uniform coatability which is free from coating defects such as comet defects, dropping defects and streak defects, even at coating using coating solution with low surface tension and low viscosity with coating die of slide hopper type, extrusion coater type or curtain coater type.

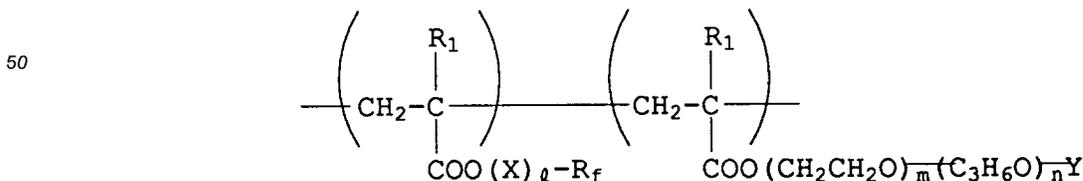
Summary of the invention

The above-mentioned object of the present invention is achieved, in case of coating by a slide hopper type coating die, an extrusion type coating die or a curtain hopper type coating die, in which (1) the coating solution is a mixture of two or more kinds of solvents having different boiling points and surface tensions, and the surface tensions are not more than 40 dyne/cm, and (2) the mixture of the solvents contains at least one surface orientating compound comprising an alkyl group, aralkyl group, aryl group or alkyl aryl group of which at least one hydrogen atom is substituted by a fluorine atom and the number of a carbon atom is 1 to 30, and (3) the added amount of the surface orientating compound is reduced from the amount of the surface tension of the solvent mixture by at least 0.5 dyne/cm.

The above-mentioned compound is a fluorine-based surfactant, and preferably contains a copolymer having recurring unit as shown in the following Formula I.

45

Formula I



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wherein, R_1 represents a hydrogen atom, a chlorine atom or an alkyl group having number of carbon atom 1 to 30. R_f represents an alkyl group, an aryl group or an alkylaryl group of which at least one hydrogen atom is substituted by fluorine atom and having a number of carbon atom 1 to 30.

Brief explanation of drawing

Figure 1

- 5 (a) Cross sectional view illustrating the oozing in extrusion coating die.
 (b) Plane view illustrating the oozing in extrusion coating die.

Detailed description of the invention

10 In general, the coater such as the slide hopper, the extrusion hopper or the curtain coater is manufactured with metal, especially the stainless steel and so on. However, since metals have large surface energy and high critical surface tension, the coating solution having low surface tension and viscosity such as organic solvent having a high wettability against metal surface is easy to spread out on the metal. Therefore, the bead cannot be formed and enough stabilized, and coating solution oozes out to the lip or peripheral part and disarranges the bead causing streak defect. The oozing solution also forms liquid drops outside of the bead, and falls on the surface of film or support causing comet defects or streak defects or edge dirt at both sides of the support.

15 The formation of liquid drops occurs remarkably when coating solution consists of two or more kinds of the mixture solvent having the different surface tension and the boiling point. The coating solution oozing from the bead among which the low boiling point component of coating solution evaporates selectively and the solvent of the high boiling point remains. When the surface tension of this high-boiling point solvent is relatively high, the solution shrinks and forms liquid drop. It is found out when the liquid drop grows up to certain size, it falls on the surface of web or support causing comet defect.

20 Moreover, when the oozing of coating solution becomes more remarkable, it spreads out of the lip and fall on the surface of coating layer, web or support causing the dropping defect.

25 Figure 1(a) is a cross sectional view which shows one example of oozing and figure 1(b) is a plane view. In the figure, 1 is a extrusion coating die, 10 is a slit where coating solution is supplied, 11 is a front bar, and 12 is a backing bar. Bead 13 is formed between web 2, running in the direction of the arrow, and front and back bar, and a coating layer is formed on the web through this bead. If the coating solution oozes at this point, bead cannot be formed completely, and instead, the oozing part 14 is formed under the regular bead. When this oozing part grows, it transfers in the web causing streak defect or the oozing part becomes liquid drop causing the dropping defect.

30 Such problems occur not only to the bead coater but also to the curtain coater. The curtain coater is the method in which the coating solution falls down like a curtain without forming bead and forms coating layer on the web by the collision power. It also has the extrusion type and the slide hopper type. In the former type, the coating solution oozes on the lip and the following slope at the slit exit where the curtain layer comes out. This oozing disturbs the separation line at the slit exit and causes streak defect on the curtain layer, or the oozing solution forms the liquid drop which gets on the curtain layer or falls on the web causing the dropping defects and the comet defects.

35 In the slide hopper type, the coating solution oozes on a beak-like lip on the point of slanting slide plane side and its back side and causes the same defects as the extrusion coater type.

It is found out that it is the most effective to add the fluorine-based surfactant which orientate on the surface of the coating solution to prevent the oozing phenomenon of such coating solution.

40 In general, the fluorine atom of the fluorine-based surfactant, e.g. perfluoroalkyl group, shows a very strong water-repellency, and is orientated supinely to the coating surface of the coating solution. When the coating solution added the surfactant is thrown into the coating die, the surfactant orientates on the surface of lip in the bead at coating and the oozing of coating solution is prevented by the water-repellent effect. It is assumed that the coating solution to which surfactant is added can prevent oozing and spread of the solution because of the autophobicity which does not get the solid surface wet by the solution itself. As a result, the streak defects, the comet defects, and the dropping defects can be prevented.

45 The oozing phenomenon of the above-mentioned coating solution occurs remarkably when the coating solution is not more than 40 dyne/cm, especially not more than 30 dyne/cm of surface tension.

50 The amount of the addition of the fluorine-based surfactant to prevent the oozing varies according to the kind of the surfactant, but it is found out that any surfactant can be used if it is added in the amount which reduces the surface tension not less than 0.5 dyne/cm.

55 Next, the fluorine-based surfactant represented by Formula I used in the present invention will be explained.

In the Formula I, R₁ includes a hydrogen atom, a chlorine atom or an alkyl groups of number of carbon atom 1 to 3, such as methyl, ethyl, and propyl, and preferable is a hydrogen atom or a methyl group.

R_f represents an alkyl group, an aralkyl group, an aryl group or an alkylaryl group of which at least one hydrogen atom is substituted by a fluorine atom, and having number of carbon atom 1 to 30, preferably 1 to 20. Preferable one among them includes perfluoromethyl group, perfluoroethyl group, perfluoropropyl group, perfluorohexyl group, perfluorooctyl group, 2,2,3,3-tetrafluoropropyl group, 2,2,3,3,4,4,5,5-octafluoroamyl group, 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptyl group, 2,2,2-trifluoroethyl group, 2,2,3,3,4,4,4-heptafluorobutyl group, 1,1,1,3,3,3-hexafluoro-2-propyl group, 1,1,1,3,3,3-hexafluoro-2-hydroxy-2-propyl group, 1,1,2,2-tetrafluoro-2-hydroxyethyl group, p-fluorophenyl group, p-trifluoromethylphenyl group, 2,3,4,5,6-pentatrifluoromethylphenyl group.

X represents



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Wherein, R₂ represents an arylene group or an alkylene group of number of carbon atom 1 to 100. L represents -O-, -S-, -N(R₃)-, -CO-, -OCO-, -SCO-, -CON(R₃)-, -SO₂-, -N(R₃)SO₂-, -SO₂N(R₃)-, and -SO-, and p represents 0 or 1. R₃ represents an alkyl group of number of carbon atom 1 to 4 such as methyl, ethyl, propyl, or butyl.

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l is 0 or an integer of 1 to 4, and preferably 0 or 1.

m is 5 to 50 and preferably 5 to 20. n is 0 or an integer of 1 to 20 and preferably 0 to 10.

Y represents alkyl group of number of carbon atom 1 to 24, and preferably is an alkyl group of a number of carbon atom 4 to 18.

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Y represents, for instance, a hydrogen atom, straight chain or branched alkyl groups such as methyl, ethyl, butyl, dodecyl, tetradecyl, and hexadecyl group, alkenyl group such as oleyl group, alkylphenyl group such as octylphenyl group or pentylphenyl group.

The molecular weight of copolymer used in the present invention is 3,000 to 200,000, and preferably between 5,000 and 50,000.

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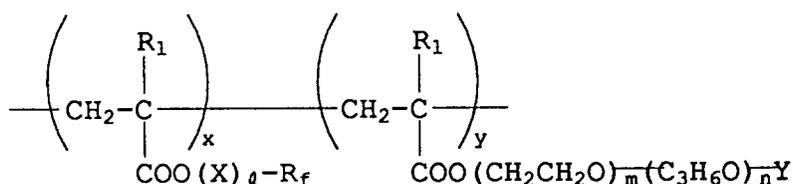
Copolymer used in the present invention must have two above comonomers as the recurring unit, but the third comonomer can be contained as long as the effect of the present invention is not ruined. The third comonomer includes, for instance, an acrylic acid alkylester, wherein the number of carbon atom of alkyl is 1 to 3, a styrene, and an ethylene.

Copolymer preferably used in the present invention is shown by the following Formula II.

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Formula II

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wherein, R₁, R_f, X, Y, l, m, and n are the same meanings as those explained in Formula I respectively.

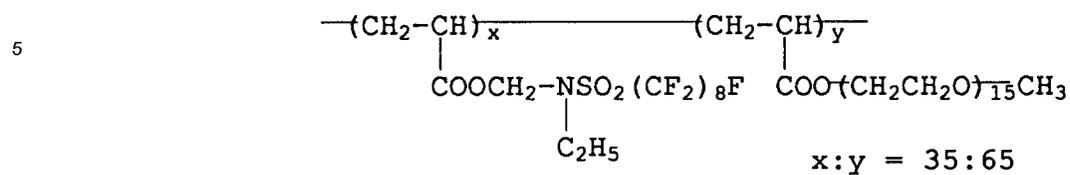
x:y = 10:90 to 80:20(mol%), and preferably 20:80 to 50:50.

Next, the concrete examples of copolymer used in the present invention are shown as follows.

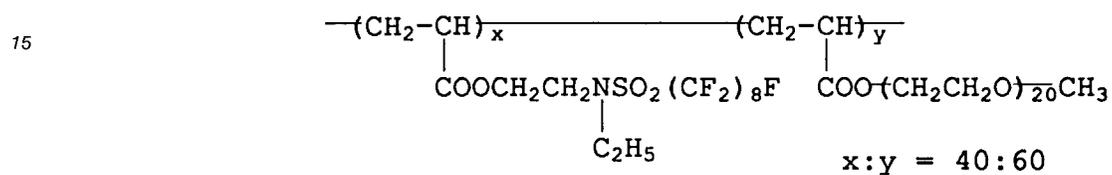
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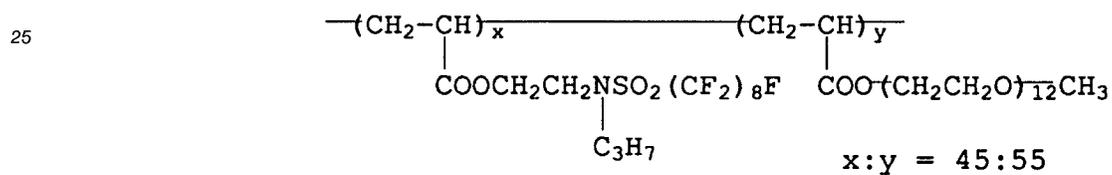
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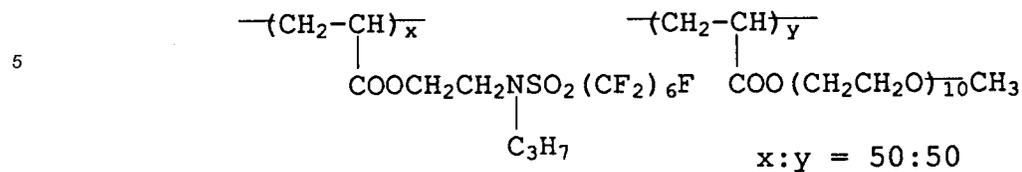
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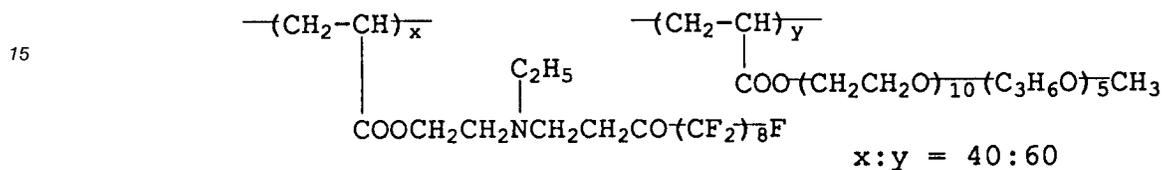


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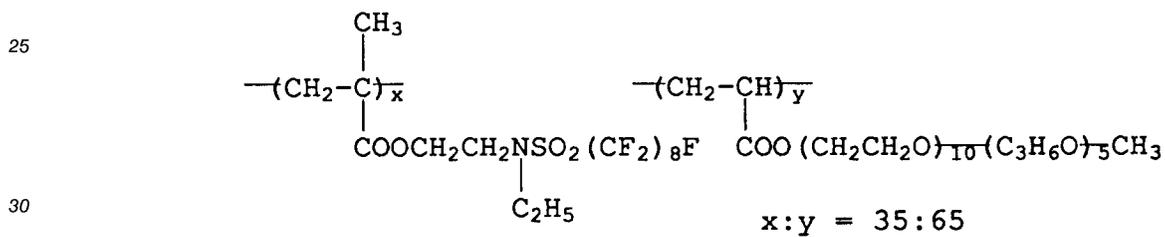
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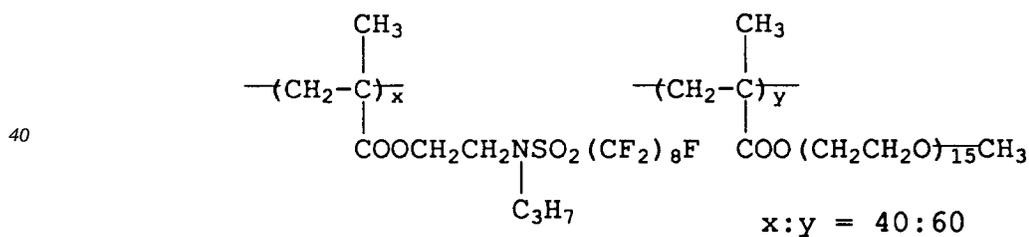
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I-7

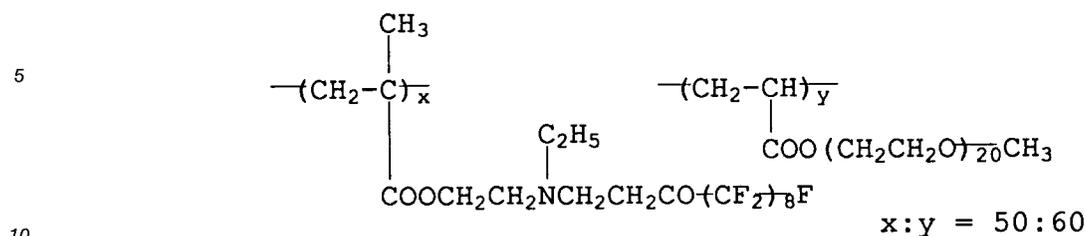


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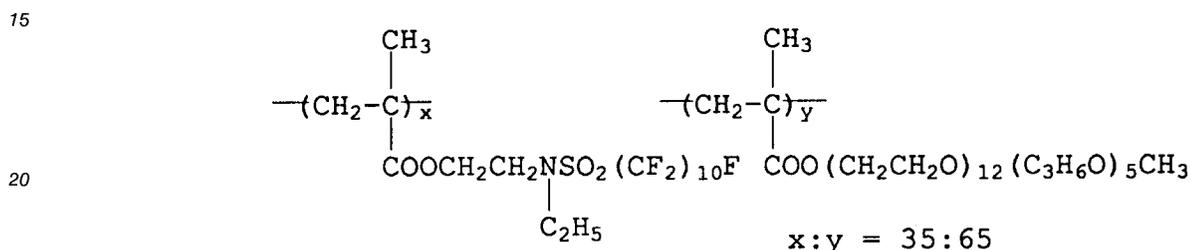
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I-8



I-9



25 In the present invention, not only above copolymer, but also the fluorine containing surfactant which has a surface orientating power against the mixture solvent used as a coating solution.

Moreover, either a water-soluble or an oil-soluble surfactant may be selected according to the solubility of used mixture solvent. Generally, the surfactant with higher solubility is preferably used for the solvent with relatively lower surface tension.

30 Fluorine-based surfactants used in the present invention and admitted the effect include those marketed in the trade names of FLUORAD FC-430 and FC-431 manufactured by U.S. 3M Co., SURFRON S-381 and S-382 manufactured by Asahi Glass Co. Ltd., and MEGAFAC F-177 manufactured by DAINIPPON INK & CHEMICALS Co. Ltd. The solvent used for the coating solution in the present invention is not especially limited, and includes, for instance, acetone, methanol, ethanol, butanol, methylglycol, isopropanol, 35 methylethylketone, methylpropylketone, cyclohexanone, toluene, cyclohexane, ethyl acetate, ethylcellulose, and methylcellulose, combination of them and water or combination of a part of them.

The terms of high boiling point component or low boiling point component, and high surface tension component or low surface tension component used in the present invention express the relative comparison of each component of the mixture solvent which uses above-mentioned solvent.

40 Among organic solvent-based, those whose boiling point is not less than 100 °C are usually handled as the high-boiling point organic solvent, and those whose surface tension is not more than 28 to 30 dyne/cm are handled as the low surface tension solvent.

When the present invention is executed, it is preferable that the temperature of coating solution and/or environmental temperature around coated part are kept to not more than 30 °C, preferably not more than 45 25 °C, for coating.

If it is 30 °C or more, the coating defect increases remarkably. For example, the oozing of coating solution on the lip increases because of the decrease of viscosity and surface tension of coating solution, or the formation of drop increases because of the acceleration of the evaporation speed caused by the vapor pressure rise of the low boiling point solvent.

50 These coating solution concretely includes photographic material layer, especially sublayer for photography, photosensitive material for printing lithographic plate, magnetic recording material, the sublayer or backing coat solution of magnetic recording material and coating solution for an electrophotographic photosensitive material and so on.

55 Examples

Hereafter, the effect of the present invention is illustrated by the example.

Example 1

The web of polyethylene terephthalate of 100 μm in thickness and 300 mm in width was subjected to the surface treatment by corona discharge and the coating solution having following formation was coated 20 ml/m² at the speed of 50 m/min, using the extrusion hopper type coating die.

(composition of coating solution)

Formation	Weight part	BP(°C)	Surface tension (dyne/cm)
Acetone	50	56.2	23.7
Methanol	25	64.5	22.5
N,N-Dimethylformamide	20	149.6	36.8
Polymethylmethacrylate	5	-	-

The surface tension of this coating solution (comparative example) was 27.2 dyne/cm, and the viscosity was 1.7 cp.

Fluorine-based surfactant FC-431, manufactured by U.S. 3M Co. was added to the above coating solution to make surface tension 26.7 dyne/cm, i.e. to reduce 0.5 dyne/cm or more of the surface tension of the coating solution. The obtained coating solution (present invention) was coated in the same coating conditions as above and following result was obtained. The numbers of each defect occurrence were shown by the number per m² of coating area.

Sample	Surface tension (dyne/cm)	Streak defect	Comet defect	Dropping defect
Inventive	26.7	0	0	0
Comparative	27.2	2 to 3	10 to 20	1 to 2

The results show that the coating method of the present invention can provide steady coating.

Example 2

Coating solution having following formation was coated 30 ml/m² by using extrusion hopper type coating die to the triacetylcellulose web of 120 μm in thickness and 1000 mm in width, at the speed of 70 m/min making decompression degree of 30 mm aq to the bead. Each coating area had 5 streak defects and 10 comet defects per m², and uniform and stable coating couldn't be obtained.

Formation	Weight part
Acetone	55
Isopropanol	25
Toluene	20
Diacetylcellulose	0.5

The surface tension of this coating solution was 24.4 dyne/cm.

Fluorine-based surfactant S-382, manufactured by Asahi Glass Co., was added to adjust to the above coating solution to make the surface tension 23.2 dyne/cm, i.e. to reduce 1.2 dyne/cm of the surface tension of the coating solution. When it was coated in the same coating conditions as above except coating speed, even and stable high speed coating was obtained up to 120 m/min of a coating rate without occurring streak defect etc..

The present invention could provide the coating method capable of obtaining uniform coatability being free from the coating defect, such as comet defect, dropping defect and streak defect even if the coating solution with low surface tension and viscosity is coated by using coating die of the slide hopper type, the extrusion coater type or the curtain coater type.

Claims

1. A method of coating using a slide hopper type, an extrusion type or a curtain hopper type coating die, in which

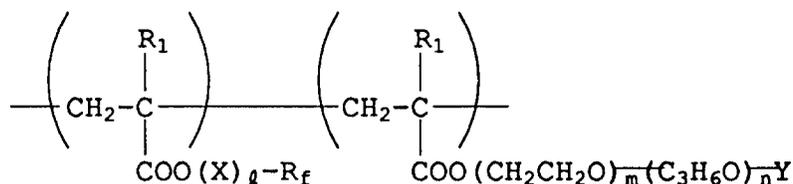
(1) the coating solution is a mixture of two or more solvents having different boiling points and surface tensions, and the surface tensions are not more than 40 dyne/cm,

(2) the mixture of the solvents contains at least one surface orientating compound comprising an alkyl group, aralkyl group, aryl group or alkyl aryl group of which at least one hydrogen atom is substituted by a fluorine atom and having a number of a carbon atom of 1 to 30,

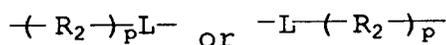
(3) the added amount of the surface orientating compound is reduced from the amount of the surface tension of the solvent mixture by at least 0.5 dyne/cm.

2. The method of claim 1, wherein the surface orientating compound is a fluorine-based surfactant.

3. The method of claim 2, wherein the surface orientating compound comprises a copolymer having recurring unit represented by formula.



wherein, R₁ represents a hydrogen atom, a chlorine atom or an alkyl group having number of carbon atom 1 to 30; R_f represents an alkyl group, an aralkyl group, an aryl group or an alkylaryl group of which at least one hydrogen atom is substituted by a fluorine atom and having a number of carbon atom 1 to 30; X represents



wherein, R₂ represents an arylene group or an alkylene group of number of carbon atom 1 to 100, L represents -O-, -S-, -N(R₃)-, -CO-, -OCO-, -SCO-, -CON(R₃)-, -SO₂-, -N(R₃)SO₂-, -SO₂N(R₃)-, or -SO-, p represents 0 or 1; R₃ represents an alkyl group of number of carbon atom 1 to 4; l is 0 or an integer of 1 to 4; m is an integer of 5 to 50; n is 0 or an integer of 1 to 20; Y represents an alkyl group of number of carbon atom 1 to 24.

4. The method of claim 3, wherein a temperature of the coating solution and an environmental thereof is not more than 30 °C.

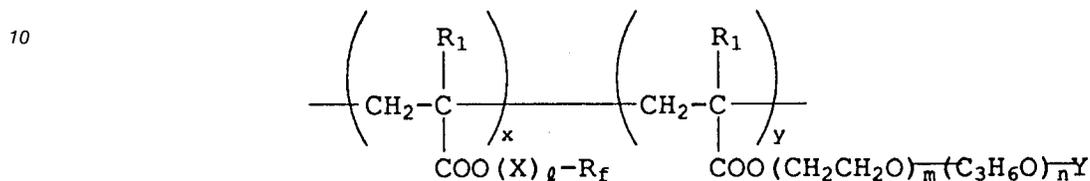
5. The method of claim 3, wherein R₁ represents a hydrogen atom or a methyl group; R_f represents an alkyl, aralkyl, aryl or alkylaryl group of which at least one hydrogen atom is substituted by a fluorine atom and having number of carbon atom 1 to 20; l is 0 or 1; m is an integer of 5 to 20; n is 0 or an integer of 1 to 10; Y represents a hydrogen atom, or an alkyl, alkenyl or alkylphenyl group.

6. The method of claim 5, wherein R_f represents perfluoromethyl group, perfluoroethyl group, perfluoropropyl group, perfluorohexyl group, perfluorooctyl group, 2,2,3,3-tetrafluoropropyl group, 2,2,3,3,4,4,5,5-octafluoroamyl group, 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptyl group, 2,2,2-trifluoroethyl

group, 2,2,3,3,4,4,4-heptafluorobutyl group, 1,1,1,3,3,3-hexafluoro-2-propyl group, 1,1,1,3,3,3-hexafluoro-2-hydroxy-2-propyl group, 1,1,2,2-tetrafluoro-2-hydroxyethyl group, p-fluorophenyl group, p-trifluoromethylphenyl group or 2,3,4,5,6-pentatrifluoromethylphenyl group.

- 5 7. The method of claim 6, wherein the copolymer is represented by formula II.

Formula II



wherein, R₁ represents a hydrogen atom, a chlorine atom or an alkyl group having number of carbon atom 1 to 30; R_f represents an alkyl group, an aralkyl group, an aryl group or an alkylaryl group of which at least one hydrogen atom is substituted by a fluorine atom and having a number of carbon atom 1 to 30; X represents

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wherein, R₂ represents an arylene group or an alkylene group of number of carbon atom 1 to 100, L represents -O-, -S-, -N(R₃)-, -CO-, -OCO-, -SCO-, -CON(R₃)-, -SO₂-, -N(R₃)SO₂-, -SO₂N(R₃)-, and -SO-

30 p represents 0 or 1; R₃ represents an alkyl group of number of carbon atom 1 to 4;

ℓ is 0 or an integer of 1 to 4;

m is an integer of 5 to 50;

n is 0 or an integer of 1 to 20;

Y represents an alkyl group of number of carbon atom 1 to 24; and a ratio of x:y is 10:90 to 80:20 in mol%.

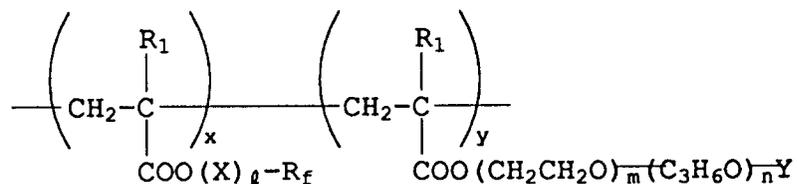
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8. The method of claim 7, wherein the ratio is 20:80 to 50:50.

9. The method of claim 3, wherein a molecular weight of the copolymer is 5000 to 50000.
- 40

10. A method of coating, which is carried under a condition of a temperature of a coating solution and an environmental thereof being not more than 30 °C, using a slide hopper type, an extrusion type or a curtain hopper type coating die, in which (1) the coating solution is a mixture of two or more solvents having different boiling points and surface tensions, and the surface tensions are not more than 40 dyne/cm, (2) the mixture of the solvents contains at least one surface orientating compound comprising an alkyl group, aralkyl group, aryl group or alkyl aryl group of which at least one hydrogen atom is substituted by a fluorine atom and having a number of a carbon atom of 1 to 30, (3) the added amount of the surface orientating compound is reduced from the amount of the surface tension of the solvent mixture by at least 0.5 dyne/cm; wherein the surface orientating compound comprises a copolymer having recurring unit represented by formula II,
- 45
- 50

Formula II



wherein, R₁ represents a hydrogen atom or a methyl group; R_f represents an alkyl, aralkyl, aryl or alkylaryl group of which at least one hydrogen atom is substituted by a fluorine atom and having number of carbon atom 1 to 20;

ℓ is 0 or 1;

m is an integer of 5 to 20;

n is 0 or an integer of 1 to 10;

Y represents a hydrogen atom, or an alkyl, alkenyl or alkylphenyl group;

X represents



wherein, R₂ represents an arylene group or an alkylene group of number of carbon atom 1 to 100,

L represents -O-, -S-, -N(R₃)-, -CO-, -OCO-, -SCO-, -CON(R₃)-, -SO₂-, -N(R₃)SO₂-, -SO₂N(R₃)-, and -SO-

p represents 0 or 1; R₃ represents an alkyl group of number of carbon atom 1 to 4;

ℓ is 0 or an integer of 1 to 4;

m is an integer of 5 to 50;

n is 0 or an integer of 1 to 20;

Y represents an alkyl group of number of carbon atom 1 to 24;

and a ratio of x:y is 10:90 to 80:20 in mol%.

FIG. 1 (a)

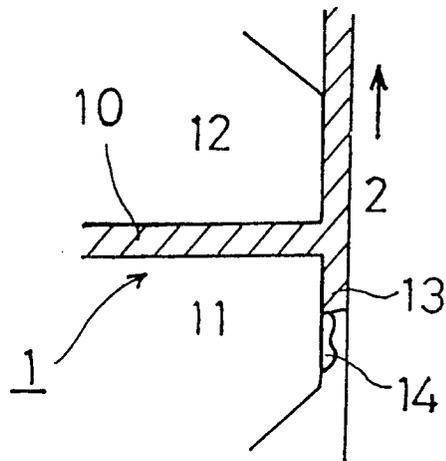
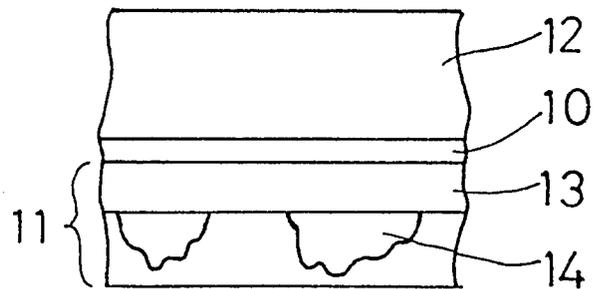


FIG. 1 (b)





European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

EP 93 10 0221

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	FR-A-2 244 788 (ICI) * page 2, line 5 - page 3, line 26 * ---	1-10	G03C1/74
Y	EP-A-0 111 338 (DU PONT) * page 2, line 23 - page 3, line 6 * * page 6, line 3 - line 20 * * page 16; table 4 * ---	1-10	
Y	GB-A-2 024 440 (FUJI) * page 2, line 18 - line 46 * * page 4, line 30 - line 36 * ---	3,5-10	
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			G03C
Place of search	Date of completion of the search	Examiner	
THE HAGUE	09 MARCH 1993	MAGRIZOS S.	
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