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(54) PROCESS FOR THE MANUFACTURE OF COATED SHEET OR WEB MATERIALS

(71) We, BEIERSDORF AG, a joint stock company organised under the laws of the Federal Republic of Germany of D-2000 Hamburg 20, Unnastrasse 48, Germany, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the manufacture of sheet or web materials which carry on at least one side an adhesive repellent layer which can be written on, particularly sheet or web pressure sensitive adhesive materials which are provided with a rear side coating which is adhesive repellent and which can be written on, as well as materials, particularly self-adhesive sheet and web materials which are obtained thereby and which are coated with an adhesive repellent coating which can be written on.

By the phrase "which can be written on" there is to be understood that the adhesive repellent layer can be written on, drawn or printed on using pencils and crayons for writing and drawing, felt tip, fibre tip and ballpoint pens using solvent containing and particularly aqueous inks and tints or printing inks, either manually or by machine.

Adhesive repellent coatings are either used in the form of carrier material of various types (slip sheets, release paper) coated therewith as a thin layer for temporarily covering the adhesive layer of self-adhesive labels or as a strengthening carrier for self-adhesive webs or foils, or they are applied on the rear side of self-adhesive tapes in order to enable these to be rolled up in reels. Adhesive repellent coatings are accordingly basically used if pressure-sensitive adhesive layers are to be preserved by an easily releasable cover or uncovered adhesive tapes are to be unrolled from a reel, even after extended storage under unfavourable conditions, with only a little effort.

Self-adhesive strips are known which can be written on and which derive this ability from the fact that the adhesive-free surface of the carrier foil is mechanically or chemically roughened or is matted by coating with a lacquer containing a high proportion of fine grain solids. Since many of the adhesive compositions which can be used have a very high adhesive power precisely to surfaces which by roughening or matting are of greatly increased area, adhesive tapes made with such compositions must generally be rolled up together with covering foils or release papers.

In order to avoid these difficulties it is already known to roughen the rear side of a carrier foil which is not to be provided with a pressure sensitive adhesive layer by mechanical or chemical means or to mat it with a lacquer containing a filler and then to provide this roughened or matted side with a thin coat of a conventional adhesive repellent material e.g. a polysiloxane containing or high molecular weight compound with long chain side groups at such a coating thickness that the rough condition of the surface is substantially retained. Such a process has the disadvantage that it requires two successive working stages, which must be relatively matched to one another satisfactorily in order to obtain a defined coating weight. This must on the one hand be small enough in order to leave free a matted surface suitable for being written on but on the other hand it must be sufficiently

thick for a satisfactory adhesive repellent effect. In industrial manufacture such requirements give rise to difficulties.

Furthermore an adhesive repellent coat is known which consists of a hot seal adhesive containing an amide wax such as e.g. a polyamino amide resin and, which receives, by the addition of matting agent, properties which are moderate so that it is capable of being written on but is still sufficiently repellent. Such a coating has the disadvantage that it adheres too well exactly to the generally preferred strongly adhesive pressure sensitive adhesives. Additionally, this coating mix yields too much under the often high pressure loadings effected by conventional writing instruments, since chemical cross-linking or hardening, which would strengthen its inner cohesiveness, is lacking. For this reason, the coating is limited to use with solvent containing inks, tints or printing pastes with which it can be written on or printed, since a mixture of the colour and the dissolved layer can take place.

Furthermore complex polyester and polyurethane polymer systems have often been proposed as adhesive repellent coatings. Under this heading there are, for example, polyvinyl fatty acid esters, polyacrylate copolymers and polyurethanes such as the reaction products of glycerine monostearate with polyisocyanates or polyvinyl carbamates. These polymers always contain components with long chain alkyls which generate a strongly anti-adhesive action and on account of their highly non-polar molecular portion also lead to a hydrophobic surface. Such adhesive repellent layers which are applied to gloss film or foil type carriers as an even flat layer thus have an exceptional separating action relative to a plurality of materials so that such surfaces can be written on with the normal writing materials such as pencil, ballpoint pen, ink or tint only insufficiently or not at all.

A polyurethane-containing adhesive repellent coat is also known from French Patent Specification 1,267,505 which can be anchored sufficiently only on paper carriers and which has a separating action only relative to customary pressure sensitive adhesives of the rubber resin type. It is obtained by the reaction of an alkyd resin with aromatic trifunctional isocyanate, which latter is the reaction product of an aromatic diisocyanate and a hexane triol. This double restriction on its ability to be used is exceptionally disadvantageous. Additionally, in this patent specification there is no direction concerning an addition of matting agent or the ability to write thereon. In contrast thereto, properties such as flexibility and softness are given as advantages of this rear side coating, which run counter to the requirements for a surface which can be written on.

In all, the known adhesive repellent coatings with organic polymer systems or polysiloxanes have the disadvantage that the degree of adhesive repellency can not be adjusted to the desired degree at all, or only with great difficulties. Thus on the one hand they have, even with the addition of a matting agent, strongly pronounced release properties so that they can only be written on in special fashion and rolls of adhesive tape provided therewith tend to fall apart as a result of too great anti-adhesive action, particularly if, pulling forces arise on rapid removal by machine. However, on the other hand, if the matting material containing layer is constituted to favour being written on, then it is insufficiently adhesive repellent and the adhesive tape can only be pulled off from the roll with difficulty. With adhesive compositions with very high adhesive power, the adhesive layer can even transfer to the rear side of the carrier.

According to a first feature of the present invention there is provided a process for the manufacture of a sheet or web material bearing on at least one side an adhesive repellent layer which can be written on, which comprises applying to at least one side of a sheet or web a layer of a solution in a solvent of, by weight per 100 parts of the total solids,

44 to 84 parts by weight of a non-reactive linear polyurethane resin which is soluble in mixtures of isopropanol with aromatics and esters, or a polyurethane resin with a hydroxypolyester content,
1 to 16 parts by weight aliphatic trifunctional isocyanate with terminal isocyanate groups,
1 to 14 parts by weight glycerin,

drying the layer and subjecting the sheet or web bearing the dried layer to a heat treatment.

The present invention also provides a sheet or web material consisting of a flexible substrate provided on one or both sides with an adhesive repellent coating

which can be written on, wherein the adhesive repellent coating is formed by reacting a mixture comprising:

44 to 84 parts by weight of an unreactive linear polyurethane resin which is soluble in mixtures of isopropanol with aromatics and esters, or a polyurethane resin with a hydroxypolyester content,
1 to 16 parts by weight aliphatic trifunctional isocyanate with terminal isocyanate groups, and
1 to 14 parts by weight glycerin,

on the substrate after application has taken place.

By the choice of suitable components and process values, and by the use of other additives in the coating, particularly siliceous matting agents and organic tin ester catalysts, a wide variety of solid adhesive repellent coatings which can be written and printed on can be produced. Such coatings are resistant to solvents, and can be well anchored to flexible sheet or web carrier materials, particularly to gloss plastics films such as for example polyvinylchloride foils. The coatings possess, relative to all pressure sensitive adhesive compositions, particularly highly adhesive types on a polyacrylate and rubber resin basis, exceptional release action. The coating is hydrophilic and absorbent in the presence of the incorporated matting agent and abrasive so that it can be satisfactorily marked by lettering and printing with all ordinary writing materials including polar liquids such as aqueous inks and indian ink. In particular, self-adhesive tapes can be made which can be written on, which can be stored, rolled up into reels and which can still be unrolled again without difficulties in use. Even after extended storage under unfavourable conditions, the initially present adhesive power of the adhesive and the particular separation action from the adhesive repellent coating remain substantially unchanged, even if plastics materials with high plasticiser contents are used as carrier sheets, such as, for example, soft polyvinylchloride.

The present invention enables the manufacture of such sheet or web materials, particularly self-adhesive materials with an adhesive repellent coat, which is simple and which can be carried out rapidly in a continuous process. The separation action of the coating and its ability to be written on are substantially independent of the application thickness of the adhesive repellent coating composition and the final drying and hardening can be carried out rapidly at low temperatures.

If desired small quantities of conventional additives and fillers can be included in the solution applied as a layer to the carrier.

As polyurethane resin, there are used according to the invention aliphatic and aromatic polyurethanes with a polymeric chain and substantially of condensed alcohol and isocyanate groups. The degree of polymerisation lies in such a range that the resin is well soluble in mixtures of isopropanol with aromatics and esters. Such polyurethane resins are normal lacquer raw materials.

The also usable polyurethane resins with a hydroxy-polyester content are co-polymers known as adhesive components and likewise having a substantially chain-like molecular construction. By a low hydroxy group content of preferably about 0.1% they are enabled to react further with isocyanates. With an average molecular weight of 150,000 to 200,000, these products are well soluble in esters and ketones and therefore well suited for the purposes of the invention.

As aliphatic tri-isocyanate any isocyanate containing terminal isocyanate groups can be used. The reaction product of 3 moles of hexamethylene diisocyanate has proved to be very advantageous. For its manufacture first 2 moles of diisocyanate are united to the corresponding urea which is then reacted with a a further mole of diisocyanate to the biuret of hexamethylene diisocyanate. The isocyanate content influences the solvent resistance, the cohesivity as well as the anchoring of the matted adhesive repellent layer to the carrier.

The release properties of the layer are dependent on the content of matting agent and on the glycerine content. By variation of the quantity of glycerin the effectiveness of the coating can be determined in simple fashion relative to the adhesive power of the pressure sensitive adhesive provided. It is also advantageous if a carrier is to be adhesive-repellent coated on both sides and the two surfaces are to have exactly defined differing release properties.

The matting agent if used may consist of finely granular siliceous compounds such as highly dispersed silicon dioxide or microfine aluminium silicate and magnesium silicate, the particle size of which is advantageously in the region of 0.1 to 15 micrometres.

Also suitable are hydrophobicised colloidal silicic acids with methylated silanol groups on the surface and particle sizes up to 30 millimicrometres.

Preferably a mixture of equal parts of silica and magnesium silicate is used, which is easy to disperse in the solution of the polymers and which has an exceptional absorbent action for thin liquid inks and tints and additionally, with a proportion of coarse grains, can act strongly abrasive relative to pencils, crayons and similar writing implements.

Preferably there are used up to 40 parts of a microfine siliceous matting agent.

If the adhesive repellent coat according to the invention contains no addition of matting agent, then because of its exceptionally hydrophilic properties it can still be written on without difficulty with aqueous inks, tints and printing inks without the liquid film breaking up and pulling together to drops because of its surface tension.

There may be employed metal organic esters as catalysts for the subsequent hardening reaction. Suitable metal organic compounds are organic tin esters such as dibutyl tin diacetate or dibutyl tin dilaurate. The organic tin esters are used in an amount of up to 1 part by weight.

As additives there can be used if desired light protection agents, anti-ageing agents, metal powders, coloured pigments or fillers.

The solvent system which is required for applying the mixture can contain, for example, one or more easily removable solvents which can be evaporated at low temperatures, such as esters, e.g. methyl, ethyl and butyl acetate, ketones such as acetone and butanone, aromatics such as toluene or xylene and alcohols such as isopropanol. If according to the invention a polyurethane with hydroxy polyester content is used, then preferably alcohol-free solvents are used, for example esters, ketones and aromatics. When using the polyurethane resin, a predominantly isopropanol-containing solvent mixture with a high proportion of aromatics and a low proportion of esters has been shown to be advantageous. Even in the presence of isopropanol, the isocyanate-containing additions show long working times.

An adhesive repellent coating can be manufactured generally as follows:

To one of the above given solvent mixtures there are added successively with intensive stirring polyurethane resin, glycerine, talcum and silicon dioxide and if desired additives, isocyanate and, if used, catalyst so that a 1—20% solution or dispersion which can be maintained is obtained. This is then directly applied using one of the conventional coating devices, for example a roller coating device, in a layer directly on to a glossy non-pretreated carrier film at such a thickness that after drying a coating weight of 0.5 to 3.5 grams/m^2 is obtained. The coating material is then fed through a drying channel, in which dwell times from a few seconds up to a minute at 60 to 90°C are sufficient, in order to remove the easily soluble solvents and carry out the hardening reaction. Subsequent cross-linking is not necessary.

Although the mechanism of the hardening reaction is not fully clear, it can be taken that both the urethane formation which is primarily to be expected between isocyanate groups and reactive hydrogen atoms of the glycerine hydroxy groups as well as, if appropriate, the hydroxy groups of the polyester containing polyurethane, arise as well as subsequent reactions of the products formed with the starting compounds, so that in all, including reaction products formed in side reactions with the water or solvent residues which cannot be eliminated, a complex elastic cross-linked polymeric reaction product forms, in which components of the mixture according to the invention are firmly enclosed not by chemical bonding.

The insensitivity of the process according to the invention relative to air humidity and impurities of the reactants is to be seen as an advantage relative to mass production, since it guarantees a satisfactory manufacture of the products according to the process of the invention.

The carrier web coated with an adhesive repellent coating on one side can then immediately be provided on the other side with a customary pressure sensitive adhesive, for example of the resin rubber-type but preferably of the acrylate variety, and rolled up to a reel. Because of the strongly adhesive repellent action, even broad webs of a flexible elastic carrier can be unrolled again without difficulty and e.g. fed to a strip cutting device.

As sheet or web carrier material all the commercial foils of plastics, modified natural materials or metals are suitable as well as papers, textiles and non-woven fabrics, on to which the adhesive repellent coating according to the invention generally adheres without difficulties, so that good possibilities of variability relative to the choice of carrier material are provided. In particular it has been

shown that glossy, chemically unpretreated, hard and soft polyvinylchloride films are well suitable, as well as polyethylene and polypropylene foils and also even polyester foils (polyethyleneterephthalate foils) as well as gloss aluminium foil.

The process according to the invention permits in simple fashion coatings which are adhesive repellent and capable of being written on without difficulty to be manufactured both with and without a matting agent included, the release properties of which are not controlled by the application thickness but by the composition of the mixture. Even with extended storage under unfavourable temperatures these separation properties remain constant since the individual components of the coating do not unmix. The coating is insensitive relative to components of the pressure sensitive adhesive and the carrier foil such as e.g. plasticisers, so that the adhesive power of pressure sensitive adhesive coatings remains the same even with long contact times between the pressure sensitive adhesive coating and the adhesive repellent coating.

The coating anchors well to practically all customary substrates, without for example glossy foil surfaces needing to be mechanically or chemically roughened, and it is elastic enough in order to adhere firmly to flexible and extensible foils. Because of these reasons it can also be used as a base layer for further coatings such as, for example, varnishes.

The adhesive repellent coating according to the invention has hydrophilic properties and, because of the matting agent particles incorporated, is additionally so absorbant that it can also be written on with highly aqueous inks and tints without difficulty. It is resistant relative to predominantly solvent containing inks and tints.

Even with hard writing implements such as ballpoints and pencils it can be written on without difficulty because of its cohesive unity and the firmly fixed abrasive agent.

The coating can be used for example for self-adhesive sticky tapes or labels which are to be written on in the form of sheets or rolls without an intermediate release paper layer or also for release sheets which are to be printed on or written on.

The adhesvie strips which can be written on likewise can be advantageously used a packaging strips and lettering foils.

The invention is further described with reference to the following specific examples:

Example 1

The following components are worked into a mixture of solvents of 50% isopropanol, 30% toluene and 20% ethyl acetate in the weight proportions given using a high speed stirrer:

59 parts by weight of linear polyurethane resin ("Desmolac 4125", ex Bayer AG)
4 parts by weight triisocyanate ("Desmodur N", ex Bayer AG)
7 parts by weight glycerin
30 parts by weight mixture of matting agents of equal parts silicon dioxide ("matting agent TK900", ex Degussa) and microtalcum.

Using the mixture so obtained of coating agents in suspension in the solvent mixture, having a solids content of 8%, a 50 micrometres thick transparent foil of hard PVC is coated on its non-pretreated glossy surface using a roller coated to a coating weight of 2.5 g/m² (dry weight).

The material provided with the reactive mixture was then heated in a drying channel of a coating plant suitable for the large surface coating of carrier materials for the purpose of drying it and reacting the coating for about 20 seconds to about 80°C.

Then there was applied to the opposite surface not provided with the coating of the carrier material a solution of a pressure sensitive adhesive based on a butylacrylate acrylic acid copolymer and containing a sucrose acetate isobutyrate as a plasticiser at a coating thickness of 25 g/m², and this was dried at 80°C.

A transparent self-adhesive material with an anti-adhesive rear side coating was obtained which could be written on exceptionally well, the unrolling force for which only amounted to about a third of the force required had the rear side been untreated.

The self-adhesive foil which can be written on so obtained can either be

immediately cut up into strips and rolled up on cores to reels ready to be sold or it can first of all be rolled up uncut and later divided into the desired sizes.

The adhesive tape rolls so manufactured retain their easy unrollability unchanged even after long storage, without any adverse effect on their other good properties. The unrolled adhesive tapes can be written on using pencil, ballpoint, inks, drawing inks and strongly aqueous inks and also using typewriter ribbon, exceptionally well. After drying the inscription on the substrate, the script can no longer be rubbed out.

Example 2

Using the working process of Example 1, on a gloss soft polyvinylchloride foil of 70 μm thickness, a reactive mixture of the following components is applied as a 20% solution in ethylacetate at a coating weight of 1.3 g/m^2 and reacted at 75°C:

80 parts by weight polyurethane resin with a hydroxy-polyester content ("Desmocoll 400", ex Bayer AG)
11 parts by weight triisocyanate ("Desmodur N", ex Bayer AG)
4 parts by weight glycerin
0.8 parts by weight dibutyl tin dilaurate
4 parts by weight hydrophobic silica ("Aerosil R 972", ex Degussa).

The further process steps were carried out as described in Example 1. The adhesive tape obtained could be written on without difficulty.

Example 3

Using the working process of Example 1 a reactive mixture of the following components as a 5% solution (suspension) in equal parts of butanone and isopropanol was coated at a coating weight of 0.8 g/m^2 on to a 25 μm thick gloss biaxially oriented polypropylene foil and reacted at 70°C:

60 parts by weight polyurethane resin ("Desmolac 4125" ex Bayer AG)
5 parts by weight triisocyanate ("Desmodur N", ex Bayer AG)
5 parts by weight glycerin
30 parts by weight talcum
0.2 parts by weight dibutyl tin diacetate

As pressure sensitive adhesive there was used a cross-linkable ethyl hexyl acrylate n-butylacrylate copolymer. A rolled up adhesive tape showed itself to be well resistant to ageing and could be written on exceptionally well with the writing devices mentioned.

Example 4

As carrier for a separating interlayer coated in adhesive repellent fashion on both sides there was used a modified paper (parchment paper) of thickness 0.6 mm and this was coated on one side with a 15% solution A and on the other side with a 15% solution B, each being a solution in equal parts of ethylacetate and butanone to a coating weight of 3.5 g/m^2, and reacted at 80°C.

Solution A

49 parts by weight polyurethane resin ("Desmocoll 400" ex Bayer AG)
8 parts by weight Triisocyanate (Desmodur N", ex Bayer AG)
7 parts by weight glycerin
35 parts by weight silicon dioxide ("Mating Agent TK900" ex Degussa)
0.5 parts by weight dibutyl tin dilaurate.

Solution B

70 parts by weight polyurethane resin ("Desmocoll 400", ex Bayer AG)
16 parts by triisocyanate ("Desmodur N", ex Bayer AG)
13 parts by weight glycerin
0.5 parts by weight dibutyl tin dilaurate.

The release properties of both sides of the release paper which can be written on manufactured in this fashion were clearly different. On the side with the smaller adhesive repellent effect there is then applied a pressure sensitive adhesive layer on a polyacrylate basis which after drying at 70°C had a coating weight of 25 g/m^2, and the finished adhesive tape was rolled up with its adhesive layer inwardly to a roll.

Such an adhesive strip according to the invention which is useful for transferring a pressure sensitive adhesive layer on to substrates of various sorts allows the possibility of marking on the uncovered side.

The words Aerosil, Desmocoll, Desmodur and Desmolac used herein are Registered Trade Marks.

WHAT WE CLAIM IS:—

1. A process for the manufacture of a sheet or web material bearing on at least one side an adhesive repellent layer which can be written on, which comprises applying to at least one side of a sheet or web a layer of a solution in a solvent of, by weight, per 100 parts of the total solids,

44 to 84 parts by weight of a non-reactive linear polyurethane resin which is soluble in mixtures of isopropanol with aromatics and esters, or a polyurethane resin with a hydroxypolyester content,
1 to 16 parts by weight aliphatic trifunctional isocyanate with terminal isocyanate groups,
1 to 14 parts by weight glycerin,

drying the layer and subjecting the sheet or web bearing the dried layer to a heat treatment.

2. A process according to claim 1 wherein the solution additionally contains up to 1 part by weight of an organic tin ester catalyst.

3. A process according to claim 1 or 2 wherein a polyurethane resin with a hydroxypolyester content having a hydroxyl content of about 0.1% is used.

4. A process according to any of claims 1 to 3 wherein a polyurethane resin with a hydroxypolyester content and having an average molecular weight of 150,000 to 200,000 is used.

5. A process according to any one of claims 1 to 4 wherein the trifunctional isocyanate is a reaction product of hexamethylenediisocyanate.

6. A process according to any one of claims 1 to 5 wherein the isocyanate has an isocyanate content of about 17%.

7. A process according to any of claims 1 to 6 wherein the solution additionally contains up to 40 parts of a microfine siliceous matting agent.

8. A process according to claim 7 wherein the matting agent consists of about equal parts of highly dispersed silica and talcum with a particle size of 0.1 to 15 μm.

9. A process according to any one of claims 1 to 8 wherein the heat treatment takes place at a temperature over 60°C, for a time of 0.1 to 1 minute.

10. A process according to claim 9 wherein the heat treatment is carried out at 80 to 90°C for about 15 seconds.

11. A process according to any one of claims 1 to 10 wherein polyvinylchloride is used as carrier.

12. A process according to any one of claims 1 to 11 wherein the sheet or web material is also coated with a strongly adherent pressure sensitive adhesive on an acrylate base on the side remote from the adhesive repellent layer.

13. A process for producing sheet or web material coated with an adhesive repellent layer substantially as hereinbefore described with reference to any of the foregoing specific examples.

14. Sheet or web material made by the process of any of claims 1 to 13.

15. A sheet or web material consisting of a flexible substrate provided on one or both sides with an adhesive repellent coating which can be written on, wherein the adhesive repellent coating is formed by reacting a mixture comprising:

44 to 84 parts by weight of an unreactive, linear polyurethane resin which is soluble in mixtures of isopropanol with aromatics and esters, or a polyurethane resin with a hydroxypolyester content,
1 to 16 parts by weight aliphatic trifunctional isocyanate with terminal isocyanate groups, and
1 to 14 parts by weight glycerin

on the substrate after application has taken place.

16. A sheet or web material according to claim 15 wherein the mixture also comprises up to 1 part by weight of an organic tin ester catalyst.

17. A sheet or web material according to claim 15 or 16 wherein the polyurethane resin used had a hydroxy-polyester content having a hydroxyl content of about 0.1%.

18. A sheet or web material according to any one of claims 15 to 17 wherein a polyurethane resin with a hydroxy-polyester content is used having an average molecular weight of 150,000 to 200,000.

19. A sheet or web material according to any one of claims 15 to 18 wherein the trifunctional isocyanate is a reaction product of hexamethylene diisocyanate.

20. A sheet or web material according to any one of claims 15 to 19 wherein the isocyanate has an isocyanate content of about 17%.

21. A sheet or web material according to any one of claims 15 to 20 wherein the mixture additionally contained 0 to 40 parts by weight microfine siliceous matting agent.

22. A sheet or web material according to claim 21 wherein the matting agent consists of approximately equal parts of highly dispersed silica and talcum with a particle size of 0.1 to 15μm.

23. A sheet or web material according to any one of claims 15 to 22 wherein polyvinylchloride is used as the flexible substrate.

24. A sheet or web material according to any one of claims 15 to 23 wherein one side of the material bears the coating of adhesive repellent material and the other side a coating of adhesive material.

25. A sheet or web material according to claim 24, wherein the adhesive material is a strongly adhering acrylate adhesive.

26. A sheet or web material substantially as hereinbefore described with reference to any one of the foregoing specific Examples.

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