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(54) **PROCESS FOR PRODUCING PRESSURE-SENSITIVE SHEET MATERIAL.**

(57) A process for producing pressure-sensitive sheet material comprising a sheet-form support having a surface coated with pressure-rupturable microcapsules, such as no-carbon required paper, pressure-sensitive adhesive sheet, etc., which process comprises coating a microcapsule-carrying side of a pressure-sensitive material with a coating solution containing defatted soybean powder prepared by treating with an alcohol soybeans defatted according to a solvent extraction process for denaturation. The coating solution containing the defatted soybean powder shows a depressed increase in viscosity of the solution upon its preparation, shows good solution fluidity and good water retention, and undergoes less changes in solution composition. In addition, it reduces unexpected rupture of microcapsules by weak pressure to remarkably decrease color stain on no-carbon required paper, pressure-sensitive paper, etc.

DESCRIPTION

METHOD OF MAKING PRESSURE SENSITIVE SHEET MATERIALS

1 TECHNICAL FIELD

This invention relates to a novel method of making pressure sensitive sheet materials. More particularly, it relates to an improvement in the method for making pressure sensitive sheet materials, such as carbonless pressure sensitive recording paper and pressure sensitive adhesive sheet, comprising a support in sheet form and, provided thereon, a coating of pressure-rupturable microcapsules.

10 BACKGROUND ART

There are known a large variety of pressure sensitive sheet materials. A typical example is a carbonless pressure sensitive recording paper utilizing the color reaction which takes place in a solvent medium between a colorless dye and a color developer such as activated clay, acid clay, phenol resins, aromatic carboxylic acids or metallic salts thereof.

Examples of carbonless pressure sensitive recording sheets include a combination type comprising, on one hand, a sheet carrying a coating formed by emulsifying a solution of a colorless dye dissolved in a solvent, encasing the emulsified finely divided liquid particles

1 in protective pellicles of natural or synthetic polymers
(i.e. microencapsulation), and coating the resulting
microcapsule suspension on a support, and, on the other
hand, a sheet comprising a support and, provided thereon,
5 a coating of color developers; and a single-layer type
comprising a support and, provided thereon, a coating
containing both the said microencapsulated colorless dye
and the color developer or microencapsulated color developer.
There is also known another combination-type carbonless
10 pressure sensitive recording paper comprising a sheet
carrying a coating of microencapsulated solution of a
color developer and a sheet carrying a coating of a
colorless dye.

Other pressure sensitive sheet materials than
15 the carbonless recording paper include a pressure sensitive
adhesive sheet carrying a coating of adhesive-containing
microcapsules or a coating of solvent-containing micro-
capsules and an adhesive, and an encapsulated perfume
sheet carrying a coating of microencapsulated perfume.

20 The carbonless pressure sensitive recording
sheet materials, which are commercially manufactured on
the largest scale of all other pressure sensitive sheet
materials, are now in use in various commodity areas such
as computer output recording sheets, business slips,
25 and business manifolded forms, and are required to have
performance characteristics which comply with the
requirements of particular uses. For this reason, in
preparing the microcapsule dispersion, various auxiliary

1 agents are added to meet various requirements. Above all,
many proposals have heretofore been made to use, as stilt
material, powdered pulp, starch powder, glass beads,
plastic beads, talc, calcium carbonate, and clays for
5 the purpose of enhancing the resistance of the micro-
capsule-coated sheet materials against those unintentional
pressing, scuffing, impact, and the like which would
cause smudging due to rupture of the microcapsules during
manufacture, handling, printing, or actual use of the
10 microcapsule-coated sheet.

Examples of proposed stilt materials for use
in carbonless pressure sensitive recording paper include
fine powders of starch and starch derivatives (Japanese
Patent Publication No. 1,178/72), starch grains having
15 an average diameter as large as at least about 1.2 times
the microcapsule diameter (Japanese Patent Publication
No. 33,204/73), corncob (waste part of an ear of corn
after removal of corn grains) [Japanese Patent Application
"Kokai" (Laid-open) No. 16,708/73], microspheres expandable
20 by heating [Japanese Patent Application "Kokai" (Laid-open)
No. 32,013/73], starch grains of large diameter derived
from beans other than soybean [Japanese Patent Application
"Kokai" (Laid-open) No. 34,013/76], acid-modified
polyolefin particles [Japanese Patent Application "Kokai"
25 (Laid-open) No. 111,810/78], water-insoluble vegetable
proteins [Japanese Patent Application "Kokai" (Laid-open)
No. 58,510/79], finely powdered polyolefin [Japanese
Patent Application "Kokai" (Laid-open) No. 51,611/79],

1 finely powdered polyolefin for use in self-contained
pressure sensitive recording paper of the single layer
type [Japanese Patent Application "Kokai" (Laid-open)
No. 3,969/80], powdered cellulose (US Patent 2,711,375),
5 and finely powdered starch (Brit. P. 1,232,347).

Stilt materials generally used for the purpose
of preventing the recording sheet from the smudging
due to unintentional rupture of the microcapsules are
finely powdered cellulose, finely powdered raw starch,
10 talc, kaolin, bentonite, pyrophyllite, and inorganic
pigments such as zinc oxide, titanium oxide, and alumina.

As described above, for the purpose of prevent-
ing the carbonless pressure sensitive recording paper from
smudging due to unintentional rupture of microcapsules
15 during manufacture, fabrication, printing, or actual use
of the recording paper, various proposals have been made
and helpful in bringing about a certain degree of improve-
ment. In particular, powdered pulp (finely powdered
cellulose) and starch grains are excellent, practical
20 stilt materials and are now in worldwide use. However,
the use of powdered pulp as stilt material involves
various problems such as a difficulty encountered in
coating operation resulting from the increase in viscosity
of the coating composition during the coating operation,
25 rendering the uniform distribution of stilt material
difficult, resulting in, on one hand, stilt-rich coating
areas, where the smudging becomes reduced, while the
color developing performance becomes deteriorated and, on

1 the other hand, stilt-poor coating areas where the smudging
becomes so enhanced that sufficient performance charac-
teristics are no more obtained from the practical view
point.

5 Therefore, an object of this invention is to
provide a method of making pressure sensitive sheet
materials which are lessened in susceptibility to the
unintensional rupture of microcapsules and the attendant
smudging, to an extent sufficient for practical use,
10 while the color developing function being retained or
improved.

 Another object of this invention is to provide
a method of making pressure sensitive sheet materials,
according to which it is possible to retard the viscosity
15 increase in the coating composition during manufacture
or application and to minimize the fluctuation in component
distribution of the coating composition in order to keep
the coating surface from deterioration in quality or to
keep the distribution of stilt materials uniform throughout
20 the coating composition.

DISCLOSURE OF THE INVENTION

 According to this invention, in making a pressure
sensitive sheet material by coating a sheet support with
pressure-rupturable microcapsules, a defatted soybean
25 powder obtained by treating and modifying defatted soybean
with an alcohol is added to a microcapsule layer-forming
composition or a composition which forms a layer contiguous

1 to the microcapsule layer. When the defatted soybean
powder obtained by treating and modifying defatted soybean
with an alcohol is used as the stilt material, the coating
composition is kept from viscosity increase during its
5 manufacture and retains desirable fluidity so as to
improve spreadability of the composition; moreover, owing
to sufficient water-retentivity of the alcohol-treated
defatted soybean powder, the fluctuation in component
distribution is kept low throughout the coating composi-
10 tion so that after application of the composition, there
is obtained a coat of uniform surface quality. For
instance, in the case of a carbonless pressure sensitive
recording paper of color developing quality at normal
level, the alcohol-treated defatted soybean powder exhibits
15 distinguished stilt effect so that the unintentional
rupture of microcapsules and the attendant smudging under
light pressing can be minimized.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The invention is described in detail in the
20 following.

The defatted soybean in powder form contains
proteins, fats, fibrous matters, ash; polysaccharides
such as galactan and pentosan; saccharides such as sucrose,
stachyose, and raffinose; and vitamins.

25 In manufacturing the defatted soybean, it is
general practice to remove, in the first step, fatty
matters from the raw soybean by expression or solvent

1 extraction. Since the removal of fatty matters by expres-
sion is insufficient, it has recently become a common
practice to use the solvent extraction which reduces
the residual fatty matter content of the defatted soybean
5 down to 1% or below. In the extraction, benzene and n-
hexane are generally used as the defatting solvent. The
treated soybean is stripped of the solvent by treating
with steam at elevated temperatures or by treating under
reduced pressure or with a solvent vapor at low temper-
10 atures.

The defatted soybean powder used in this
invention is obtained from the solvent-extracted soybean
by treating with alcohols such as methanol, ethanol, and
propyl alcohol. If the defatted soybean produced by
15 expression is used as stilt material, owing to the
residual oil which amounts to about 8%, there will occur
in the coating layer a phenomenon of cissing which
hinders uniform distribution of the stilt material. The
defatted soybean powder used in this invention is that
20 obtained from the solvent-extracted soybean by treating
and modifying with an alcohol to adjust the protein
content to 45 to 55%, the water-soluble nitrogen content
being 5 to 15% of the total nitrogen content. Such a
defatted soybean powder is an effective stilt material
25 and, in addition, has an advantage of improving the
water retentivity of the capsule-containing coating
composition on account of the presence of water-soluble
constituents. If the water-soluble nitrogen content

1 exceeds 15% of the total nitrogen, gelation will take
place in the coating composition, whereas if the content
is below 5%, the water retention becomes insufficient.
The principal object of treating and modifying the defatted
5 soybean with an alcohol is deodorizing and decoloring.
The resulting defatted soybean powder is a powder material
which is nonsticky, easy to handle, and pale amber yellow
in color. As a consequence, the coating layer containing
such a stilt material is excellent in surface brightness.

10 A powder material prepared by dissolving raw
soybean or defatted soybean in an alkali solution and
reprecipitating with an acid is generally a high-purity
(e.g. 85%) protein substance and substantially devoid of
water-solubility, the water-soluble nitrogen content
15 being 1% of the total nitrogen or less. For this reason,
as ascertained by the present inventors, such a soybean
powder is unsuitable for actual use, because if it is
added to a capsule-containing coating composition, the
composition becomes less uniform in component distribution,
20 as compared with the composition prepared according to
this invention.

The defatted soybean powder used in this inven-
tion is amorphous in particle shape and has an average
particle size of from 10 to 60 μm , especially preferred
25 size being 20 to 30 μm which are larger than the capsule
size, though not limitative. If necessary, the defatted
soybean powder can be used in combination with other known
stilt materials such as, for example, finely powdered

1 cellulose or wheat starch to obtain also a desirable
result.

Typical examples of the processes for producing
microcapsules used in this invention are physical,
5 coacervation, interfacial polymerization, and in situ
polymerization processes.

In the case of carbonless pressure sensitive
recording paper, typical examples of colorless dyes enclosed
in the capsules include phthalides such as 3,3-bis(4-
10 dimethylaminophenyl)-6-dimethylaminophthalide and 3,3-
bis(1,2-dimethylindol-3-yl)-5-dimethylaminophthalide;
fluoranes such as 3-diethylamino-6-methyl-7-anilino-
fluorane, 3-(N-methylcyclohexylamino)-6-methyl-7-anilino-
fluorane, 3-diethylamino-6-methyl-7-chlorofluorane, 3-
15 diethylamino-6-methyl-7-chlorofluorane, and 3-diethylamino-
7-dibenzylaminofluorane; thiazine compounds such as benzoyl-
leuco methylene blue; lactams such as N-(p-nitrophenyl)-
rhodamine B lactam; spiro compounds such as 1,3,3-
trimethylindolinospiropyrene; and indolyl red. As for
20 the pellicle materials for microcapsules, there may be
mentioned, nonlimitatively, unmodified or partially
modified natural polymers such as gelatin, cellulose
derivatives, and starch derivatives; and synthetic resins
such as urea-formaldehyde resin, melamine-formaldehyde
25 resin, reaction products of isocyanate compounds and
hexamethylenediamine, and reaction products of adipic acid
dichloride and hexamethylenediamine. However, in view
of the reinforcement of capsule wall properties such as

1 heat resistance, solvent resistance, water resistance,
chemical resistance, and impact resistance, microcapsules
covered with synthetic resins are preferred.

As oily substances which constitute the internal
5 phase of microcapsules, there may be used solvents of high
boiling points which can be at least one selected from
natural oils such as petroleum- or mineral-base oils, and
animal- or vegetable-base oils or from synthetic oils.
Especially preferred are those which dissolve the colorless
10 dyes. As examples, mention may be made of alkylated
biphenyls, alkylated terphenyls, alkylated naphthalenes,
triarylmethanes, diarylalkanes, phthalic esters, phosphoric
esters, sulfonic esters, diaryl ethers, and higher alkyl-
benzenes, but the invention is not limited thereto.

15 The pressure sensitive sheet material manu-
factured according to this invention comprises at least
a support, microcapsules, and a defatted soybean powder
obtained from the solvent-extracted soybean by treating
and modifying with an alcohol. In addition to the micro-
20 capsules and the said defatted soybean powder, the present
sheet material may contain binders including water-soluble
natural binders such as starch and carboxymethylcellulose;
and water-soluble synthetic binders such as polyvinyl
alcohol, polyvinylpyrrolidone, and polyacrylic acid; and
25 latices such as styrene-butadiene latex, styrene-butadiene-
acrylic acid latex, butadiene rubber latex, and neoprene
rubber latex. Further, if necessary, there may be added,
as lubricant or extender, inorganic pigments such as talc,

1 titanium dioxide, zinc oxide, calcium carbonate, and
activated clay; and organic lubricants such as ethylene-
bistearamide.

The coating composition of this invention can
5 be applied by any of the known techniques such as, for
example, air knife coating, blade coating, bill blade
coating, roll bar coating, three-applicator-roll coating,
and curtain coating. In general, the blade coating is
effectively used in applying a high-solid content coating
10 composition. The coating composition of this invention
can be efficiently applied also by the blade coating
technique, because even at high solids concentration, it
is hardly subject to fluctuation in distribution of compo-
nents and has excellent fluidity.

15 The defatted soybean powder and the microcapsules
should be present on the same side of the support. There-
fore, it can be added to the coating composition which is
to form the microcapsule layer, or to the coating composi-
tion which is to form an under- and/or over-layer to the
20 microcapsule layer.

The composition of solvent-extracted soybean
is generally described as follows: about 50% or less of
crude protein, 25 to 30% of carbohydrate, 3 to 5% of crude
fiber, 5 to 6% of ash, and 1% or less of crude fat.
25 Accordingly, one half is protein substances and another
half is non-protein substances. According to this invention,
defatted soybean containing a certain amount of water-
soluble proteins produces a good result. As examples of

1 commercial products, mention may be made of "S-Sanmeat"
(Ajinomoto Co.) extra grade (7.0% of water, 54.0% of
protein, and 0.2% of oil) and first grade (7.5% of water,
49.0% of protein, and 0.2% of oil).

5 The invention is illustrated below in detail
with reference to Example, but the invention is not limited
thereto. Hereinafter all parts are by weight.

EXAMPLE (Alcohol-treated defatted soybean powder was
used.)

10 Example of microcapsule preparation:

	Parts
Crystal Violet Lactone	4
Benzoyl Leuco Methylene Blue	1
3-Diethylamino-6-methyl-7-anilino-fluorane	0.5
Diarylethane-base organic solvent ("Hi-Sol SAS", Trademark, produced and supplied by Nippon Petroleum Chemical Co.)	100

The above solution of an electron donating leuco
dye in the high-boiling solvent was emulsified in 100
parts of a 5-% aqueous solution of a styrene-maleic acid
copolymer. An aqueous solution of melamine-formaldehyde
15 prepolymer was prepared by heating a mixture (adjusted to
pH 9.5 with sodium hydroxide) of 10 parts of melamine,
25 parts of 37-% formalin, and 20 parts of water. The
prepolymer solution was added to the above emulsion and
allowed to react with stirring at 75°C for 90 minutes.
20 The reaction mixture was cooled to room temperature

1 and adjusted to pH 9.5 with sodium hydroxide to obtain a
microcapsule dispersion. To 100 parts (dry basis) of the
dispersion, was added 35 parts of a defatted soybean
poder ("S-Sanmeat" extra grade, a product of Ajinomoto
5 Co.; a soybean powder produced by treating solvent-
defatted soybean with an alcohol), 35 parts of talc, 17
parts of polyvinyl alcohol, and 17 parts (dry basis) of
a styrene-butadiene latex. The mixture was thoroughly
stirred to form a dispersion. The dispersion was coated
10 by means of an air knife coater on a plain paper (40 g/m²
in basis weight) at a coverage of 5 g/m² (dry basis) to
obtain an upper sheet for a carbonless pressure sensitive
recording paper.

COMPARATIVE EXAMPLE 1 (Wheat starch was used.)

15 An upper sheet was obtained in the same manner
as in Example, except that 35 parts of wheat starch was
used in place of the defatted soybean powder treated with
an alcohol.

COMPARATIVE EXAMPLE 2 (Finely powdered cellulose was used.)

20 An upper sheet was obtained in the same manner
as in Example, except that 35 parts of finely powdered
cellulose was used in place of the defatted soybean powder
treated with an alcohol.

COMPARATIVE EXAMPLE 3 (No stilt material was used.)

25 An upper sheet was obtained in the same manner

1 as in Example, except that the defatted soybean powder
treated with an alcohol was omitted and each 13 parts of
polyvinyl alcohol and a styrene-butadiene latex were used
as binder for 100 parts of the capsules, the coverage with
5 respect to capsules being the same as in Example.

COMPARATIVE EXAMPLE 4 (Defatted soybean not treated with
an alcohol was used.)

An upper sheet was obtained in the same manner
as in Example, except that 35 parts of solvent-defatted
10 soybean (50% in protein content) not treated with an
alcohol was used in place of the defatted soybean powder
treated with an alcohol.

COMPARATIVE EXAMPLE 5 (Water-insoluble soybean protein
powder was used.)

15 An upper sheet was obtained in the same manner
as in Example, except that 35 parts of a high-purity
soybean protein powder having a water-insoluble protein
content of about 85% or above was used in place of the
defatted soybean powder treated with an alcohol.

20 Color developer sheet:

A pressure sensitive recording sheet (Mitsubishi
NCR paper CF) carrying a coating of an electron accepting
solid acid (oil-soluble novolak-type phenol resin) was
used as color developer sheet (under sheet). The upper
25 sheet was placed on the under sheet so as to bring both

- 1 coated sides in contact with each other and tested for coloring characteristics and smudge characteristics. The test results were as shown in the table. The test results for smudge characteristics were as shown in the table.

Table

	Coloring intensity (D intensity)		Smudge (reflectance) (%)		Viscosity of coating composition (cps)		Fluctuation of component distribution of coating composition
Example	0.50	○	83.7	○	19.0	○	○
Comp. Example 1	0.46	Δ	89.8	○	13.5	○	x
" 2	0.50	○	83.5	○	59.0	x	Δ
" 3	0.52	○	66.8	x	15.0	○	○
" 4	0.49	○	69.2	x	120.5	x	○
" 5	0.50	○	83.9	○	15.0	○	x

Note for mark:

- Excellent for practical use.
 Δ Unsatisfactory for practical use.
 x Unsuitable for practical use.

1 In the table, the coloring intensity is the
intensity of color measured after one hour of pressure
application at 90 kg/cm by means of a calender. The
smudge is expressed in terms of reflectance measured 24
5 hours after rubbing under a load of 450 g/cm³. All
numerical values were corrected for the background condi-
tion. Marks, ○ and X, were the results of visual
inspection performed at the same time.

 As is apparent from the table, a recording
10 paper system comprising a powder material obtained by
treating and modifying the solvent-defatted soybean with
an alcohol showed especially well-balanced practical
characteristics, being excellent in coloring characteristics,
less subject to smudging, low in viscosity of the coating
15 composition, and less subject to the fluctuation of compo-
nent distribution in the coating composition.

POSSIBILITY OF INDUSTRIAL APPLICATION

 As described in the foregoing, the method of
this invention is suitable for the production of carbon-
20 less pressure sensitive recording paper, pressure sensitive
adhesive sheet, perfume-containing capsule sheet, and the
like. Above all, it is especially suited for the produc-
tion of pressure sensitive recording paper, because it
keeps the recording paper from smudging due to uninter-
25 national rupture of the microcapsules.

SCOPE OF CLAIMS

1. In a method of making a pressure sensitive sheet material by coating pressure-rupturable microcapsules on a sheet support the improvement which comprises providing
5 on the side of the support carrying said microcapsules a coating layer containing a defatted soybean powder produced by treating and modifying the solvent-extracted soybean with an alcohol.
2. A method of making a pressure sensitive sheet
10 material according to Claim 1, wherein the defatted soybean powder has a protein content of 45 to 55% and the water-soluble nitrogen content is 5 to 15% of the total nitrogen content.
3. A method of making a pressure sensitive sheet
15 material according to Claim 1, wherein the defatted soybean powder has an average particle size of 10 to 60 μm .
4. A method of making a pressure sensitive sheet material according to Claim 1, wherein the coating composition which provides the coating layer containing a
20 defatted soybean powder is obtained by adding said defatted soybean powder to a microcapsule-containing coating composition.
5. A method of making a pressure sensitive sheet material according to Claim 1, wherein the coating composition which provides the coating layer containing the
25 defatted soybean powder contains no capsule and is coated over and/or under the microcapsule layer.

INTERNATIONAL SEARCH REPORT

0144430

International Application No. PCT/JP84/00248

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) *		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. ³ B41M5/12		
II. FIELDS SEARCHED		
Minimum Documentation Searched *		
Classification System	Classification Symbols	
IPC	B41M5/12-5/22	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched *		
Jitsuyo Shinan Koho 1960-1983 Kokai Jitsuyo Shinan Koho 1971-1983		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴		
Category ¹⁵	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸
A	JP, A, 51-34013 (A. E. Staley Manufacturing Co.) March 23, 1976 (23.03.76) & US, A, 3,996,060	1
* Special categories of cited documents: ¹⁴ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search ²	Date of Mailing of this International Search Report ²	
August 6, 1984 (06.08.84)	August 13, 1984 (13.08.84)	
International Searching Authority ¹	Signature of Authorized Officer ²⁰	
Japanese Patent Office		