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<p>(54) Title: HYBRID RESIN EMULSIONS</p> <p>(57) Abstract</p> <p>Water-based adhesive coating applications where high-speed coating processes are desired have improved mechanical strength by using as tackifier water-based emulsions containing blends of hydrocarbon resins with minor amount of rosins.</p>		

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Hybrid Resin Emulsions

Technical Field

This invention relates to water-based adhesive coating applications such as for pressure sensitive adhesive compositions and products containing water-based resin emulsions having improved mechanical stability under high shear conditions, and in particular to adhesives based on water-based emulsions of blends of hydrocarbon resins with minor amounts of rosins.

Background of the Invention

It is known that one of the largest uses for hydrocarbon resins is in adhesives. These adhesives are frequently classified by the form in which they are applied to the substrate, ie, solvent, hot-melt or aqueous emulsion. In addition, adhesives are classified by markets, eg, packaging, construction, industrial, pressure-sensitive, transportation, and consumer. Pressure-sensitive adhesives ("PSA") are typically used in coating applications for or onto any of a number of substrates, and aqueous emulsions are used in many such applications, eg, tapes, labels and films.

The behaviour of aqueous emulsions containing hydrocarbon resins in coating operations, particularly in high-speed coating operations, is often characterized by mechanical instability of the emulsion. By mechanical instability is meant the tendency for the formation of grit or coagulum when the emulsions are submitted to mechanical shear forces. Under the coating condition this leads to deposits at or in critical locations within coating equipment. Excessive formation of the grit or coagulum can cause non-uniform coating and can lead to frequent equipment shut-downs for cleaning. Grit or coagulum formation results from the coalescence of emulsified particles when the emulsion is submitted to mechanical shear forces.

The high shear stress occurring during high-speed coating operations, or during any other processing or handling operations of adhesive systems containing the hydrocarbon resin emulsions, can lead to particle coalescence, and grit or coagulum formation. In particular, coating of adhesive emulsions can be carried out by several methods. The most commonly used methods for coating of pressure-sensitive adhesives are reverse roll and knife-over-roll. In addition, gravure, wire-wound rod, floating knife, air knife, blade and other methods are also used. Several of these methods (eg, knife and blade) are known to be

high shear methods, see Handbook of Pressure-Sensitive Adhesive Technology, ed. by D. Satas, Ch. 26, pp. 498-532 (Van Nostrand Reinhold Co., 1982).

The high shear mechanical stability of hydrocarbon resin emulsions has been addressed in EP-A-0 240 253. This document describes resin emulsions suitable for PSA in high-speed coating lines to reduce deposits and extended coating times. The hydrocarbon resin emulsions are prepared by forming a water-in-oil aqueous emulsion of unsaturated material reactive with the hydrocarbon resin, causing a reaction between the hydrocarbon resin and the reactive material, and adding sufficient water to invert the emulsion to one of reacted hydrocarbon resin in water.

Additionally, patent documents address improving the shelf-like stability of hydrocarbon resin or rosin emulsions and/or hydrocarbon resin and rosin mixtures said to be suitable generally for adhesive applications. US-A-4 477 613 addresses an aqueous base tackifier of improved shelf stability characterized by the presence of a resinous tackifier composition comprising a carboxyl containing rosin having an acid number value from 30-150 and containing a counter ion capable of rendering it soluble or dispersible in water, a minor amount of surfactant and a minor amount of an elastomeric latex. The resinous tackifier composition is defined as a rosin or rosin derivative, such as one prepared by esterification, having unreacted carboxyl attached. Optionally, up to 90% of the total tackifier solids may be a compatible resinous tackifier selected from low acid value rosin or rosin derivative, hydrocarbon petroleum resins, or polyterpene resins. An abstract for JP-A-55-025 444 addresses aqueous emulsions for paper sizing, paint, ink or adhesive preferably containing 1-50wt.% hydrocarbon resin and 50-99wt.% esterified product of selected rosin plus rosin modified with α,β -unsaturated carboxylic acids. Alternatively the hydrocarbon resin is an esterified product of acid-modified petroleum hydrocarbon resin. Storage stability is said to be provided resins containing as emulsifier a) a water-soluble polycarboxylic acid, b) a water-soluble salt of a rosin acid, and c) at least one compound of the formula $R'(OR'')_nOH$. The use of such resins or emulsions thereof in or for high speed processing is not addressed. United Kingdom patent 1324488 is also concerned with blend of rosins and resins for paper sizing.

In conducting tests of high speed coating operations with hydrocarbon resin emulsions blended with polyacrylic emulsions in order to assess the utility of such resins for this application, applicant discovered a significant problem with grit and/or coagulum arose.

Accordingly the high shear strength and high adhesion to non-polar surfaces provided by hydrocarbon resins in PSA composition could not be effectively utilized.

Attempts were then conducted to mix hydrocarbon resin emulsions with rosin acid emulsions for use in high speed coating operations, but again significant grit or coagulum occurred.

It has been found that a water-based adhesive of polymer emulsions and a water-based resin emulsion which an emulsified blend of a) hydrocarbon resin and a minor part of b) a rosin, rosin derivative, or blends thereof, has improved mechanical stability under high shear conditions. By blending a) and b) components and emulsifying the resulting blend, the resulting hybrid resin emulsion is exceptionally suitable for use with polymer emulsions in water-based adhesives used in high-speed coating applications where high shear stresses are likely to be developed.

The hydrocarbon resin used are the known low molecular weight thermoplastic polymers derived from cracked petroleum distillates, turpentine fractions, coal tar, and a variety of pure monomers. The number average molecular weight is usually below 2000, and physical forms range from viscous liquids to hard, brittle solids. Polymerization feedstreams are derived from the sources described above via various known means and methods of fractionation. Friedel-Crafts polymerization is generally accomplished by use of appropriate catalysts in a polymerization solvent, and removal of solvent and catalyst by washing and distillation. Thermal polymerization is also utilized to some extent, particularly for aliphatic and aliphatic-aromatic petroleum resins. The preferred hydrocarbon resins are those known to be useful as tackifiers for adhesive compositions, particularly the petroleum resins derived from the deep cracking of petroleum distillates, hydrocarbon resins from pure aromatic monomers, the coumarone-indene resins from coal tar and the polyterpenes derived from turpentine fractions. Included in petroleum resins are those that have been modified with aromatic or terpene containing feedstreams. For additional description of feedstream derivation, monomer composition, and methods of preparation reference may be made to patent literature (see Background) and to technical literature, eg. *Encycl. of Poly. Sci. and Eng'g.*, vol. 7, pp. 758-782 (John Wiley & Sons, 1987). In particular, reference may be made to EP-A-O 240 253; its corresponding application U.S. ser. no. 07/065,792, filed 24 June 1987, is incorporated by reference for purposes of U.S. patent practice.

It is known that many of the above hydrocarbon resins may be hydrogenated and/or modified by subsequent chemical reaction. Hydrogenation is typically accomplished under pressure in the presence of a metal catalyst. Acid-modified hydrocarbon resins may be prepared in accordance with the process described in EP-A-O 240 253, or in accordance with the descriptions in the prior art documents listed therein. Such hydrogenated and modified hydrocarbon resins, particularly those of the petroleum resins, are additionally suitable in accordance with the invention. Additionally, mixtures of any of the foregoing will also be suitable in accordance with the invention.

Hydrocarbon resin products, including unmodified, modified and fully or partially hydrogenated, are widely available. For example purposes reference can be made to the Escorez[®] hydrocarbon resins available through Exxon Chemical Belgium, and Exxon Chemical Co., U.S.A.; the Arkon[®] hydrocarbon resins available from Arakawa Chemical, Japan; the Petrosin[®] hydrocarbon resins available from Toho Petroleum, Japan ; the Regalite[®], Regalrez[®], Endex[®], Kristalex[®], Piccolastic[®], Piccotex[®], Hercures[®], Piccopale[®], Piccotac[®], Picco[®], Piccourmaron[®], Hercotac[®], and Adtac[®] hydrocarbon resins of Hercules Inc., U.S.; the Zonatac[®], Zonarez[®], hydrocarbon resins available from Arizona Chemical Co., U.S.A ; the Wingtack hydrocarbon resins available from Goodyear Chemicals, U.S.A ;and the Eastotac hydrocarbon resins available from Eastman Kodak Co., U.S.A. Particularly preferred petroleum hydrocarbon resins for use in this invention will include those described in EP-A-O 196 844, specifically those having a softening point from 10°C to 120°C and being copolymer of a feed comprising C₅ olefins and diolefins and from 10 to 60 wt. % monovinylaromatic compound.

The rosin used include any of those known in the art to be suitable as tackifying agents, and includes preferably those that have been modified by hydrogenation,

disproportionation or dismutation, fortification, dimerization or polymerization (including those that have been partly or fully esterified). The modified rosins and rosin derivatives are preferred. The rosin acids are particularly suitable for compositions of the invention. Mixtures of any of the foregoing will also be suitable. The principal sources of the rosin include gum rosin, wood rosin, and tall oil rosins which typically have been extracted or collected from their known sources and fractionated to varying degrees. Additional background can be obtained from technical literature, e.g., *Kirk-Othmer Encycl. of Chem. Tech.*, vol. 17, pp. 475-478 (John Wiley & Sons, 1968) and Handbook of Pressure-Sensitive Adhesive Technology, above, pp. 353-356. Rosin and rosin derivative products suitable in accordance with the invention are available commercially, for example, Foral[®], Pentalyne[®], and Staybelite[®] of Hercules, Inc., U.S.; and Sylvatac[®] of Sylvachem Corp., U. S.; Bevitack[®], Bevilite[®], Bevipale[®] of Bergvick Kemi AB, Arizona Chem. Co., U.S.; Gresinox[®], Dertoline[®], Dertopoline[®], Granolite[®], Hydrogral[®], Dertopol[®], Polygral[®], of DRT S.A., France ; Oulutac[®] of Oulu OY, Sweden ; Tergum[®], Terlac[®], Tergraf[®] of Resinas Sinteticas S.A., Spain ; and ; Residis[®] of Union Resinera Espagnola S.A., Spain.

By "minor amount" of rosin or rosin derivative it is meant 50 wt.% or less of the total hybrid resin is the rosin component, in other words, the hydrocarbon resin represents 50 wt.% or more of the total resin components. As little as 15 wt.% rosin component results in significant improvement in the mechanical stability of resins prepared with suitable emulsifiers, see below, some noticeable improvement results from inclusion of even less. Though more rosin component than hydrocarbon resin component can be effectively used, it is no longer properly termed a "hydrocarbon resin emulsion", and takes on properties more close to those of a rosin emulsion. Thus the rosin component will preferably constitute 10 up to 50 wt.% or less, and more preferably 10-45 wt.%, most preferably 10-25 wt.%.

The method of preparation of the hybrid resin emulsion of the invention in its broadest terms includes the steps of blending the a) hydrocarbon resin and a minor part of the b) rosin or rosin derivative and emulsifying the resulting blend. Preferably the hydrocarbon resin and rosin components are melt-blended so as to achieve an intimate blend and are then emulsified in any manner suitable for preparing aqueous emulsions or dispersions. Typically the emulsification process will be by direct emulsification or inverse emulsification, by either of batch or continuous processes. A preferred continuous, direct emulsification process comprising the use of static mixers is described in EP-A-0 283 247, and its corresponding application U.S. ser. no. 07/547,534 filed 29 June 1990. An alternative inversion process is described in US-A-4 414 346. Both of the latter are

incorporated by reference for purposes of U.S. patent practice. General background on emulsions and their preparation, including equipment, may be found in the technical literature, e.g., *Kirk-Othmer Encycl. of Chem. Tech*, vol. 8, pp. 900-930 (John Wiley & Sons, 3d ed., 1979) and *Encycl. of Emulsion Tech.* (Marcel Dekker, Inc., 1979).

The preferred blending comprises blending the hydrocarbon resin and rosin components by any physical means, typically in a heated, stirred vessel. The resin and rosin components of the invention should preferably be molten or liquid under the blending conditions employed. Alternatively, softening to a point that the components are effectively blended is sufficient. In many cases the hydrocarbon resin or rosin component of higher softening or melting point will be at least partly soluble in the lower softening or melting point component, or one or the other, or both, will be liquid, any of which permits of the intimate blending preferred. Temperatures necessary to achieve appropriate softening or solubilising of the components are achieved by the energy of mixing and/or by heating. The necessary temperatures depend upon the softening points of the components and can be determined empirically within ordinary polymer engineering practices.

It is known that in an emulsion, the larger the dispersed phase particle, the greater is the tendency for phase separation. Thus fine particles promote shelf stable emulsions. The selection of suitable emulsifiers, increasing the energy input during emulsification, or thickening the continuous aqueous phase are all known to allow decreased particle size and improved shelf-life stability of the emulsion and can be applied with this invention.

In particular the emulsification process step will include providing a surfactant as emulsifier. Any of the known emulsifier compositions suitable for rosin emulsions are suitable in accordance with the invention, but most preferably the anionic or non-ionic surfactants are used. Preferred examples of the anionic surfactants include those derived from polyoxyethylene, like alkyl- or alkylaryloxy-poly(ethyleneoxy)-ethanol-sulfates, sulfonates, sulfosuccinates and phosphates. Preferred non-ionic surfactants are those derived from polyoxyethylene like alkyl- or alkylaryloxy-poly(ethyleneoxy)-ethanol. Typically the amount of emulsifier used will be from 2-10 pbw per 100 parts hydrocarbon resin, more typically 4-7 pbw. Additionally, it was observed in the preparation of the examples that follow, that increasing the length of the hydrophilic polyethylene oxide chain of the preferred surfactants further improved the mechanical stability. Likely this occurs from increasing the steric barrier to particle coalescence.

It is believed that the inclusion of the rosin component with the hydrocarbon resin compound increases the polarity of the emulsified material, and the energy needed to overcome the repulsion between the dispersed phase particles. As a result coalescence in a significant degree is avoided, even under higher stress conditions previously thought impractical for hydrocarbon resin emulsions. Typically the high speed coating operations used for PSA compositions can achieve roll-speeds of up to about 300 m/min, have a tolerance between roller and blade of 10-40 micron(micrometer) and accordingly exhibit a shear rate on the order of approximately 10^5 sec^{-1} . By use of the invention petroleum resins can be used in such operations without the previously observed formation of significant coagulum or grit.

In a preferred embodiment the adhesives are water-based PSA compositions that are applied by a high speed coating process to a typical substrate such as thermoplastic film, siliconized paper for transfer coating for the production of tapes, labels and the like. In such PSA compositions the polymer emulsion that is blended with the hybrid emulsion of the invention are any of those known for PSA applications and will include those based on polyacrylics (particularly the rubbery polyacrylates) and modified polyacrylics ; natural rubber and blends thereof with synthetic elastomers ; styrene block polymers ; styrene-butadiene rubber and carboxylated derivatives ; and butyl rubber (including modified butyl copolymers containing α -methyl or ρ -methyl styrene, halogenated butyl and modified butyl polymers, polyisobutylene, and mixes).

Further background may be found in technical literature, for example, Chs 10-15 of Handbook of Pressure-Sensitive Adhesive Technology, above, and in patent literature, see for example EP-A-O 196 844 and its corresponding U.S. ser. no. 07/058,652 filed 21 May 1987, the disclosures of which are incorporated by reference for purposes of U.S. patent practice. Typical wt.% of polymer emulsion component in PSA is 50-90, based on total dry weight of the PSA composition.

The following examples are presented to illustrate the foregoing discussion. All parts, proportions and percentages are by weight unless otherwise indicated. Although the examples may be directed to certain embodiments of the present invention, they are not to be viewed as limiting the invention in any specific respect.

Examples

The following examples demonstrate preparation of hybrid emulsions and the resulting mechanical stability as measured by Test A and Test B below.

The hydrocarbon resins were made by the Friedel-Crafts copolymerization of C₅ olefins and C₅ diolefins petroleum feedstocks with styrene according to EP-A-O 196 844 and its corresponding U.S. ser. no. 07/058,652, incorporated by reference above.

The resins had the following characteristics:

HC1: 50°C softening point	30 wt % styrene
HC2: 73°C softening point	30 wt % styrene

The hydrocarbon resins were blended with rosins and the blends then emulsified via an inverse emulsification process as described in US-A-4 414 346. The blending step involved the melting and the blending of the hydrocarbon resin with the rosin(s) in a stirred reactor within a temperature range of 80°C to 95°C., prior to the surfactant addition and the inverse emulsification stage. The resulting emulsions were characterized for particle size with a Coulter N4 Submicron Particle Size Analyzer (results in nanometers, "nm").

The mechanical stability of the resulting hydrocarbon resin emulsion was evaluated by blending the hydrocarbon resin emulsion with an acrylic emulsion, and submitting the blend to mechanical shear forces. Comparison with the mechanical stability of commercially available rosin emulsions (currently used in high speed coating applications) in the same test is provided.

The usefulness of the hybrid emulsions in waterbased adhesive applications has been evaluated against commercial rosin emulsions in the tackification of waterbased acrylic emulsions for pressure sensitive adhesive applications.

Mechanical stability

Test A

The resin emulsion was mixed with NeoTac[®] 576 (acrylic emulsion of ICI Resins Ltd., U.K., having a solids content of 63 wt %) and diluted with deionized water such as to

result in an emulsion blend characterized by a resin/acrylic dry weight ratio of 20 / 80 and a total solids content of 56 wt %.

12.5 gr of the emulsion blend was placed in a 100 cc glass beaker with a magnetic bar and a cover and stirred on a magnetic stirrer for 3 hours at 1000 RPM. The resulting mixture was then diluted with water and the coagulum formed was collected, wash and dried. Table 1 below reports the amounts of coagulum formed expressed in wt % of the total solids of the emulsions.

Test B

The resin emulsion was mixed with the same acrylic emulsion as in test A such as to result in a hydrocarbon resin/acrylic dry weight ratio of 30 / 70 and a total solids content of 56 wt %. 150 gr of the emulsion blend was placed in a cylindrical flask with a few drops of defoamer (Drewplus[®] 4510, Drew Ameroid N.V., Belgium). The mixture was sheared with a Ultraturrax T 25 rotor stator for 5 minutes at 8000 RPM while the temperature was maintained at 22 °C. The coagulum formed in the test was collected and dried. The amount formed is reported in the tables below as wt % of the total solids of the emulsions.

Table 1

Starting resins HC resin/rosin	Resin wt ratio	Surfactant (wt %) (1)		emulsion part. size	Mechanical stability (wt % coagulum)	
					Test A	Test B
HC1	100	Atlox 3404 FB 6%		250 nm	7.5	1.1
HC1/RWW	90/10	CO 433	6%	311 nm	4.8	n
HC1/RWW	75/25	CO 433	6%	236 nm	4	n
HC1/RWW	50/50	CO 433	6%	230 nm	1.6	n
RWW	100	CO 433	6%	205 nm	0.6	n
HC2	100	Atlox 3404 FB 6%		255 nm	7.2	1.2
HC2/RWW	90/10	CO 433	6%	265 nm	3.7	n
HC2/RWW	75/25	CO 433	6%	184 nm	2.3	0.74
HC2/RWW	50/50	CO 433	6%	190 nm	1.8	0.33
HC1/SRE	75/25	CO 433	6%	192 nm	n	0.45
HC1/SRE	75/25	EP 115	6%	223 nm	n	0.21
HC1/SRE	75/25	EP 120	6%	228 nm	0.4	0.05

(1) in wt % active on total hydrocarbon resin plus rosin weight

Notes: n - not measured

Atlox[®] 3404FB : alkyl benzene sulfonate and non ionic surfactant blend provided by ICI Specialty Chemicals, Belgium ;

Antarox[®] CO 433 : sodium salt of nonlyphenoxy (polyethyleneoxy) ethanol sulfate having 4 methylene oxide units

Fenopon[®] EP 115/ EP 120 : ammonium salt of nonylphenoxy (polyethyleneoxy) ethanol sulfate having chain length respectively = 20 & 30 ethylene oxide units provided by G.A.F. Belgium, N.V. ;

RWW : wood rosin (soft. pt = 75 °C) provided by Hercules BV, Holland;

SRE : Staybelite[®] resin E hydrogenated rosin acid manufactured by Hercules BV, Holland (soft pt = 75 °C);

Note : The pure hydrocarbon resins could not be emulsified with the same surfactants as the hybrid resins nor could the reverse be done owing to resins polarity/surfactant HLB incompatibilities. Thus surfactants useful for rosin/rosin derivative emulsions were used for the hybrid resins.

Table 2 : Multicomponent hybrid resin emulsions

Hybrid emulsion resin composition (wt %)					Mechanical Stability (wt % coagulum)
HC1	SRE	SE10	SE3	code No	test B
85	15	0	0	H1	0.05
70	20	10	0	H2	0.10
70	20	0	10	H3	0.04
55	25	10	10	H4	0.06
55	15	20	10	H5	0.07
Permatac [®] A 751					0.42
Permatac [®] A 522					0.08
Snowtac [®] 8/21 CF					0.33

(all hybrid emulsions made with Fenopon EP 120 - 5 wt% active surfactant on total hydrocarbon resin plus rosin weight)

SE 10 : Staybelite[®] Ester 10 - Glycerol ester of hydrogenated rosin acid
(soft. pt. = 85 °C) manufactured by Hercules BV, Holland

SE 3 : Staybelite[®] Ester 3 - triethylene glycol ester of hydrogenated rosin acid

(liquid resin) manufactured by Hercules BV, Holland

Permatac[®] : rosin emulsions provided by Alliance Technology Products, Ltd., U.K.;

Snowtac[®] : rosin emulsions provided by Tenneco Malros Ltd., U.K.

Table 3 : PSA properties of hybrid resin emulsions

Resin emulsion	Peel strength (FTM 1)* (N/inch)		Loop tack (FTM 9)* (N/inch)		Shear (FTM 8)* (Hrs)
	steel	PE ¹	steel	PE ¹	1" x 1" x 1 Kg
H1	15	8.8	14.8	7	12.5
H2	15.8	7.8	15.5	8	17.6
H3	15.5	8.6	15	9	16.5
H4	15.3	8.6	18.5	8.9	7.4
H5	13.6	8.1	14.2	8	11.5
Permatac [®] A751	17.4	8	16.5	7	9.3
Permatac [®] A522	16.2	8	16.8	7.7	11

1) PE was untreated polyethylene cast film (ESCORENE[®] LD 151, Exxon Chemical Co., U.S.A.), 200 micron (micrometer)

Note: properties were evaluated on blends of resin emulsion with NeoTac[®]576 acrylic emulsion at a dry weight ratio of 30 : 70 and coated on Mylar[®] (E.I. DuPont de Nemours, Inc., U.S.A.) and measured according to FINAT TESTING METHODS.

The properties exhibited by these hybrid resin emulsions saw them to be particularly useful in water based adhesive.

The following is claimed:

Claims:

1. A water-based adhesive composition comprising an emulsified blend of a) hydrocarbon resin and b) a minor part of a rosin or rosin derivative, or blends thereof and a polymer emulsion wherein said polymer is one or more of polyacrylics (particularly the rubbery polyacrylates) and modified polyacrylics; natural rubber and blends thereof with synthetic elastomers ; styrene block polymers ; styrene-butadiene rubber and carboxylated derivatives ; and butyl rubber (including modified butyl copolymers containing α -methyl styrene, halogenated butyl and modified butyl polymers, polyisobutylene, and mixes).
2. The adhesive composition of claim 1 wherein the b) component is a rosin acid and constitutes from 10 to 25 wt % of said emulsified blend.
3. The resin emulsion according to claim 1 or claim 2 wherein said resin is petroleum resin having a softening point from 10°C to 120°C and being a copolymer of a feed comprising C₅ olefins and diolefins and from 10 to 60 wt % monovinylaromatic compound.
4. The use of a resin emulsion comprising emulsified blend of a) hydrocarbon resin and b) a minor part of a rosin or rosin derivative, or blends thereof in waterbased adhesive applications for the purpose of improving mechanical stability under high shear stress conditions.