



US 20060137700A1

(19) **United States**

(12) **Patent Application Publication**
Gong et al.

(10) **Pub. No.: US 2006/0137700 A1**

(43) **Pub. Date: Jun. 29, 2006**

(54) **TRIACETIN RESISTANT HOT MELT
ADHESIVE**

Publication Classification

(76) Inventors: **Lie-zhong Gong**, Bridgewater, NJ
(US); **Ingrid Cole**, Flemington, NJ
(US); **Abhi Narthana**, Belle Mead, NJ
(US)

(51) **Int. Cl.**

A24D 3/06 (2006.01)

A24B 15/42 (2006.01)

(52) **U.S. Cl.** **131/332; 131/331**

Correspondence Address:
National Starch and Chemical Company
10 FINDERNE AVENUE
BRIDGEWATER, NJ 08807 (US)

(57)

ABSTRACT

(21) Appl. No.: **11/023,052**

(22) Filed: **Dec. 27, 2004**

Hot melt adhesives comprising a polar tackifier are triacetin resistant and particularly advantageous for use as a plug wrap sealing adhesive. Particularly preferred for use in the practice of the invention are hot melt adhesives comprising a rosin ester tackifier.

TRIACETIN RESISTANT HOT MELT ADHESIVE

FIELD OF THE INVENTION

[0001] The invention relates to hot melt adhesives that are particularly useful in plug wrap sealing applications. The hot melt adhesives of the invention have excellent triacetin resistance.

BACKGROUND OF THE INVENTION

[0002] Cigarette filters are typically made from a crimped textile tow of cellulose acetate fibers. The textile tow is first bloomed to separate the cellulose acetate fibers, and then an organic solvent, more specifically triacetin, is applied to soften the fibers. The bloomed textile tow is then formed into a tube, and wrapped in paper (plug wrap) to form cigarette filters. The plug wrap paper is glued together along on longitudinal edge with a hot melt adhesive. In many instances, the constructed filter plug is actually multiple lengths of filter, which is then cut and separated into the individual filters later in the cigarette manufacturing process, i.e., prior to joining the tobacco rod to the filter plug.

[0003] Resistance to triacetin is a key parameter to be considered in the selection of a hot melt adhesive for plug wrap sealing applications. This is because during storage, triacetin migrates to the glue line and weakens the adhesive bond, resulting in a shorter shelf life of the resulting cigarette filters. There is, therefore, a need in the art for adhesives having improved triacetin resistance that can advantageously be used in plug wrap sealing applications. The invention addresses this need.

SUMMARY OF THE INVENTION

[0004] The invention provides the art with a hot melt adhesive having excellent resistance to triacetin and filter plugs prepared using the adhesive and cigarettes comprising such filters. It has been found in accordance with the present invention, that hot melt adhesives comprising polar tackifiers provide unexpected advantages in cigarette filter manufacture.

[0005] One embodiment of the invention is directed to a cigarette filter comprising a hot melt adhesive composition containing a polar tackifier, preferable a rosin ester, most preferably a light color rosin ester.

[0006] Another embodiment of the invention is directed to a method for preparing a cigarette filter comprising applying hot melt adhesive comprising a polar tackifier to plug wrap paper, rolling the plug wrap around a filter plug and bonding the laps together with said adhesive. Preferably the polar tackifier is a rosin ester, most preferably a light color rosin ester.

[0007] Yet another embodiment of the invention is directed to a cigarette comprising a filter made using a hot melt adhesive comprising a polar tackifier, preferable a rosin ester, most preferably a light color rosin ester.

DETAILED DESCRIPTION OF THE INVENTION

[0008] Cigarettes are typically constructed using four adhesive applications; these involve the "tow anchor," the filter seam, the "rod" side seam, and the "tipping."

[0009] In a typical cigarette construction application, there are two separate processes: the filter making, and the rod and cigarette construction.

[0010] In the first process, the filter material (usually cellulose, acetates, or other fibrous materials) is formed into the "plug," or the body of the filter. Adhesive is then applied to hold the filter plug in place on the plug wrap paper; this adhesive application is known as the "tow anchor." The plug wrap is then rolled around the filter plug and the laps bonded with another adhesive, to form the "filter seam." The filter is then sent for further processing. In many applications, the constructed filter plug is actually multiple lengths of filter, which can then be cut and separated into the individual filters necessary later in the application.

[0011] In the second process in the manufacture of cigarettes, the tobacco rod is formed by placing and shaping the tobacco on the side seam paper, and then joining the rod to the filter plug and creating the finished cigarette. The side seam paper (usually fed from a web) is wrapped around the tobacco and adhesive applied to the laps, creating the rod. Sections of the rod can then be cut and touched to the filter plug, and both of them bonded together by placing tipping paper and adhesive around the junction. The finished cigarettes are then processed and collated for packaging.

[0012] At present, hot melt adhesives used commercially for filter or plug wrap sealing application are polyethylene and ethylene vinyl acetate based hot melt adhesives. These are preferred since they are relatively non-polar and provide some resistance to the deteriorative effects of triacetin (glyceryl triacetate or 1,2,3-propanetriol triacetate). C₅ and C₉ hydrocarbon tackifiers and alpha methyl styrene are generally chosen because such tackifiers are relatively non-polar and therefore have some resistance to triacetin, which is relatively polar.

[0013] The invention provides hot melt adhesives comprising a particular type of tackifier that have surprising resistance to triacetin. It has now been discovered that use of a hot melt adhesive comprising a polar tackifier, such as rosin ester, in the manufacture of cigarette filters offer much better resistance to triacetin than hot melt adhesives currently in commercial use. While not being bound to any particularly theory, it is believed that hot melt adhesives comprising polar tackifier such as rosin ester possesses better interaction to the plug wrap paper, leading to improved resistance to triacetin.

[0014] The present invention is directed to the use of hot melt adhesive compositions in the manufacture of cigarette filters. In particular, the present invention is directed to a process of making cigarette filters, and to cigarette filters, wherein a hot melt adhesive comprising a rosin ester tackifier is applied to at least one longitudinal edge or surface of porous and/or non-porous plug wrap paper. One embodiment of the invention is directed to a method of making a cigarette filter comprising wrapping a plug wrap paper having a leading and trailing end around the filter plug such that the leading and trailing ends overlap and securing the overlapping ends with a hot melt adhesive comprising a polar tackifier. The adhesives of the present invention are also applicable to multi-component filters or other filters which are prepared by e.g., preapplying the hot melt to filter paper, then heating the coated paper on the filter making machine.

[0015] The present invention is directed to the manufacturing process, and to cigarette filters, which comprise a hot melt adhesive comprising a rosin ester tackifier, and to cigarettes prepared using filters comprising a rosin ester tackifier.

[0016] Hot melt adhesives suitable for use herein preferable will comprise at least one ethylene copolymer. Examples include polyethylene, ethylene n-butyl acrylate copolymers, ethylene/ α -olefin copolymers, and ethylene vinyl acetate copolymers and well as mixtures and blends thereof.

[0017] The ethylene n-butyl acrylate copolymers (EnBA) useful herein will generally contain at least about 10 to 40 weight percent n-butyl acrylate and have a melt index of at least about 40 dg/min, preferably at least about 400 dg/min. The preferred copolymers are available from Exxon under the designation XW 23-AH and comprise approximately 33 to 37 weight percent n-butyl acrylate by weight and have a melt index of about 400 dg/min. The amount of the copolymer present in the adhesive varies from about 5 to 45 weight percent by weight, preferably about 10 to 40 weight percent by weight. Mixtures of EnBA copolymers may also be used. The adhesive compositions of the present invention may optionally comprise a second EnBA copolymer, specifically one containing about 30 to 35, preferably 33 weight percent by weight of n-butyl acrylate and having a melt index of about 6 to 40 dg/min. The preferred copolymers are available from Elf Autochem under the designation LOTRYL 35BA40 and contain approximately 35 weight percent n-butyl acrylate by weight and have a melt index of about 40 dg/min. The amount of this copolymer present in the adhesive varies from about 1 to 25 weight percent by weight, preferably 5 to 20% by weight.

[0018] Ethylene/ α olefin polymers useful herein will generally have a composition distribution breadth index greater than 50%, and Mw/Mn less than 6, a melt index of 40 to 1000 dg/min, a melt point of 71 to 90° C., a density of 0.850 to 0.92. As used herein, composition distribution index, or short chain branching distribution index, refers to the weight percent of polymer molecules having a comonomer content within 50% of the median total comonomer content. The ethylene/ α olefin polymers may be copolymers of ethylene and any α olefin, for example butene.

[0019] The ethylene vinyl acetate copolymers (EVA) useful herein are those containing at least about 15 to 45 weight percent by weight vinyl acetate and having a melt index of at least about 6 dg/min and up to about 500 dg/min. The EVA copolymers will preferably comprise less than 40 weight percent vinyl acetate (VA), most preferably 28%-35% VA. The amount of the copolymer present in the adhesive varies from about 5 to 45 weight percent by weight, preferably about 10 to 40 weight percent by weight. Mixtures of ethylene vinyl acetate copolymers may also be used.

[0020] The adhesive may also comprise a polymeric additive selected from the group consisting of ethylene methyl acrylate polymers containing 10 to 28 weight percent by weight methyl acrylate, ethylene acrylic acid copolymers having an acid number of 25 to 150, methyl(meth)acrylate copolymers, polyethylene, polypropylene, poly(butene-1-co-ethylene)polymers and low molecular weight and/or low melt index ethylene n-butyl acrylate copolymers. When such additive is present, it is present in amounts up to about 15 weight percent by weight of composition.

[0021] The adhesive of the present invention will comprise one or more tackifying resins. The tackifiers are present in the adhesive compositions of the present invention in an amount of 10 to 60 weight percent by weight of the composition, preferably 20 to 40 weight percent. Whereas prior art hot melt adhesives used in filter manufacturing comprise a non-polar tackifier such as C₅ and C₉ hydrocarbon tackifiers and alpha methyl styrene, the adhesive used in the practice of the current invention comprises a polar tackifier. By polar tackifier is meant a tackifier having ester groups, hydroxyl groups or acid groups. Examples of such polar tackifier is terpene phenolic tackifiers and rosin ester tackifiers. Rosin esters are particularly well suited for use in the practice of the invention and are preferred.

[0022] It has been discovered that hot melt adhesives comprising rosin ester as tackifier has improved triacetin resistance. While typical grades of rosin ester have a yellow color (color>3 in solid), and therefore are not highly desirable for the plug wrap application since in many cases the plug wrap paper is white, new grades of rosin esters with extra light color (color<3 in solid) are now commercially available from several companies such as Arizona Chemical (e.g., Sylvalite RE 105 XL) and Hercules (e.g., Pexalyn 6110) and are preferred for use in the practice of the invention. Hot melt adhesives using these extra light color rosin esters possesses light color and therefore are preferred for applications of sealing plug wrap.

[0023] Waxes may be usefully employed in the adhesive compositions of the present invention. Waxes are commonly used to modify the viscosity and reduce tack at concentrations up to 60% by weight, preferably less than about 45% by weight. Waxes useful in the adhesives of the present invention include paraffin waxes, microcrystalline waxes, Fischer-Tropsch, polyethylene and by-products of polyethylene wherein M_w is less than 3000. More preferably, the concentration of wax is less than 35% by weight for high melt point waxes.

[0024] Paraffin waxes suitable for use in the present invention include those having melting points in the range of about 130 to 200° F. (54° C. to 193° C.), such as, for example, PACEMAKER from Citgo, and R-2540 from Moore and Munger; and low melting point synthetic microcrystalline or Fischer-Tropsch waxes having a melting point of less than about 180° C. The most preferred wax is paraffin wax with a melting point of 150° C.

[0025] The adhesives of the invention preferably also contain a stabilizer or antioxidant. Among the applicable stabilizers or antioxidants included herein are high molecular weight hindered phenols and multifunctional phenols such as sulfur and phosphorous-containing phenol. Hindered phenols are well known to those skilled in the art and may be characterized as phenolic compounds which also contain sterically bulky radicals in close proximity to the phenolic hydroxyl group thereof. In particular, tertiary butyl groups generally are substituted onto the benzene ring in at least one of the ortho positions relative to the phenolic hydroxy group. The presence of these sterically bulky substituted radicals in the vicinity of the hydroxyl group serves to retard its stretching frequency, and correspondingly, its reactivity; this hindrance thus providing the phenolic compound with its stabilizing properties. Representative hindered phenols

include; 1,3,5-trimethyl 2,4,6-tris(3,5-di-tert-butyl-4-hydroxybenzyl)benzene; pentaerythrityl tetrakis-3(3,5-di-tert-butyl-4-hydroxyphenyl)propionate; n-octadecyl-3(3,5-di-tert-butyl-4-hydroxyphenyl)-propionate; 4,4'-methylenebis(2,6-tert-butylphenol); 4,4'-thiobis(6-tert-butyl-o-cresol); 2,6-di-tertbutylphenol; 6-(4-hydroxyphenoxy)-2,4-bis(n-octyl-thio)-1,3,5 triazine; di-n-octylthioethyl 3,5-di-tert-butyl-4-hydroxybenzoate; and sorbitol hexa[3-(3,5-di-tert-butyl-4-hydroxyphenyl)-propionate].

[0026] Commercially available antioxidants include the hindered phenols known as IRGANOX, and available from Ciba-Geigy.

[0027] The performance of these antioxidants may be further enhanced by utilizing, in conjunction therewith known synergists such, for example, as thiodipropionate esters and phosphites, particularly useful is distearylthiodipropionate. These stabilizers, if used, are generally present in amounts of about 0.1 to 1.5 weight percent, preferably 0.25 to 1.0%. Other additives such as plasticizers, pigments, dyestuffs conventionally added to hot melt adhesives for various end uses contemplated as well as small amounts of additional tackifiers and/or waxes such as paraffin wax may also be incorporated in minor amounts, i.e., up to about 10% by weight, into the formulations of the present invention.

[0028] Small amounts of additional tackifiers and/or waxes such as microcrystalline waxes, hydrogenated castor oil, amide waxes and vinyl acetate modified synthetic waxes may also be incorporated in minor amounts, i.e., up to about 10 weight percent by weight, into the formulations of the present invention.

[0029] The adhesive compositions are prepared by blending the components in the melt at a temperature of about 121° C. until a homogeneous blend is obtained, approximately two hours. Various methods of blending are known to the art and any method that produces a homogeneous blend is satisfactory.

[0030] The present invention also contemplates that the adhesives may be a blend of any of the above described polymers or copolymers. For example, the base polymer in the adhesive may be a blend of EnBA and EVA polymers. Other blends include polyethylene and EVA blends, and poly α -olefin and EVA blends. These and any other combination of polymers could be used as the base adhesive, with the additional ingredients chosen according to the polymers selected.

[0031] The hot melt adhesive for use in the practice of the invention can be formulated for application at either high temperature (>300° F.) or lower temperature (200-300° F.).

EXAMPLE

[0032] All adhesive formulations were prepared using a single blade mixer at temperature of 300° F. The adhesives were then subjected to the following tests.

[0033] Viscosity of hot melt adhesives was measured using a Brookfield Thermasel viscometer with a number 27 spindle. The Gardner color of the hot melt adhesive was measured at 250° F., at which the adhesive was molten.

[0034] Adhesive bonds were made using a ¼ inch bead width on non-porous plug wrap paper substrates with dimension of 2 inch wide and 3 inch long. Adhesion was determined by aging the adhesive bonds at various temperature for 24 hr and then separating the bonds immediately. The resulting percentage of fiber tear (FT) was recorded.

[0035] In determination of triacetin resistance, the adhesives bonds were soaked in triacetin for 24 hr at ambient temperature and then were separated immediately. The relative bond strength and the resulting fiber tear were recorded. Triacetin resistance is determined to be good if more than slight fiber tear is observed and moderate to good bond strength is obtained using the tests described above.

[0036] Several adhesive formulations were prepared as described above. The formulations of these adhesives are set forth in Table 1, where Example 1 to 4 uses rosin ester as tackifier, and Example 5 uses a blend of rosin ester and alpha methyl styrene as tackifiers.

TABLE 1

Raw material		Ex 1	Ex 2	Ex 3	Ex 4	Ex 5
IRGANOX 1010	Anti-oxidant	0.5	0.5	0.5	0.5	0.5
PACEMAKER 53	paraffin wax	30	30	30	30	28
Kristalex 3085	alpha methyl styrene					17
SYLVALITE RE 110L	Rosin ester				32	
Sylvalite RE100L	Rosin ester			30		
ELVAX 210W	EVA 28/420			30	28	
ELVAX 240	EVA 28/43	10	24	10		10
ESCORENE 7840S	EVA 33/43				10	
EVATANE 33-400	EVA 33/400	28	14			28
Sylvalite RE 105 XL	Rosin ester	32	32			17

[0037] Triacetin resistance of the adhesives shown in Table 1 was determined as described above. The results are shown in Table 2. Adhesive Examples 6 and 7 are conventional adhesives containing hydrocarbon tackifier that are currently used commercially for plug wrap sealing.

TABLE 2

	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6	Ex 7
Application temperature	275° F.	315° F.	275° F.	275° F.	275° F.	350° F.	250° F.
Viscosity @ application temp (cps)	2110	2135	2370	2365	2290	2550	2500
Gardner Color at 250° F.	1+	1+	3+	4	1+	2-	2-

TABLE 2-continued

	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6	Ex 7
<u>Adhesion on non-porous plug wrap paper</u>							
FT % at room temp.	75%	90%	50%	75%	70%	80%	70%
FT % @ 40° F.	80%	100%	40%	40%	65%	65%	60%
FT @ 120° F.	55%	90%	40%	70%	50%	70%	65%
<u>Triacetin resistance</u>							
FT in triacetin	yes	yes	yes	yes	slight	no	no
Bond strength in triacetin	moderate	strong	moderate	moderate	weak	weak	weak

[0038] As shown in Table 2, all adhesives showed good adhesion (good Fiber Tear (FT)) from room temperature to 120° F. In terms of triacetin resistance, Example 1 to 4 showed FT and good bond strength in triacetin, while Example 5, Example 6, and Example 7 gave slight FT or no FT and weak bond strength in triacetin. The results demonstrated that Example 1 to 4 had better triacetin resistance than the currently commercially available hot melt adhesives for sealing plug wrap applications. Example 1 & 2 are particularly preferred embodiments because their excellent triacetin resistance and their lower Gardner color value.

1. A cigarette filter comprising a hot melt adhesive composition containing a polar tackifier.

2. The cigarette filter of claim 1 wherein the polar tackifier is a rosin ester.

3. The cigarette filter of claim 1 wherein the adhesive comprises at least one ethylene copolymer.

4. The cigarette filter of claim 3 wherein the adhesive comprises an ethylene vinyl acetate copolymer.

5. The cigarette filter of claim 4 wherein the adhesive comprises from about 20 to about 45 wt % ethylene vinyl acetate and from about 25 to 40 wt % of a rosin ester tackifier.

6. The cigarette filter of claim 1 wherein the rosin ester has a Gardner Color at 250° F. of less than about 3.

7. The cigarette filter of claim 6 wherein the rosin ester has a Gardner Color at 250° F. of less than about 2.

8. A method of making a cigarette filter comprising wrapping a plug wrap paper having a leading and trailing end around the filter plug such that the leading and trailing

ends overlap and securing the overlapping ends with a hot melt adhesive comprising a polar tackifier.

9. The method of claim 8 wherein the polar tackifier is a rosin ester.

10. The method of claim 8 wherein the adhesive comprises at least one ethylene copolymer.

11. The method of claim 10 wherein the adhesive comprises an ethylene vinyl acetate copolymer.

12. The method of claim 11 wherein the adhesive comprises from about 20 to about 45 wt % ethylene vinyl acetate and from about 25 to 40 wt % of a rosin ester tackifier.

13. The method of claim 8 wherein the rosin ester has a Gardner Color at 250° F. of less than about 3.

14. The method of claim 13 wherein the rosin ester has a Gardner Color at 250° F. of less than about 3.

15. A cigarette comprising the filter of claim 1.

16. The cigarette of claim 15 wherein the polar tackifier is a rosin ester.

17. The cigarette of claim 16 wherein the adhesive comprises an ethylene vinyl acetate copolymer.

18. The cigarette of claim 17 wherein the adhesive comprises from about 20 to about 45 wt % ethylene vinyl acetate and from about 25 to 40 wt % of a rosin ester tackifier.

19. The cigarette of claim 15 wherein the rosin ester has a Gardner Color at 250° F. of less than about 3.

20. The cigarette of claim 15 wherein the rosin ester has a Gardner Color at 250° F. of less than about 2.

* * * * *