

Heat-sealing compound for sealing aluminium foil and polyethylene terephthalate film to polypropylene, polyvinyl chloride and polystyrene containers

5 Field of the invention

The tablets and foodstuffs packaging sector has for some time now seen the entry of polypropylene (PP) and polyethylene terephthalate (PET), as well as
10 polystyrene (PS) and polyvinyl chloride (PVC). Unpretreated polypropylene is a very critical plastic for the coatings sector, since very often there arise problems with adhesive strength. Although these problems take on a somewhat different perspective when
15 heat sealing to propylene is being carried out (PP in this case is not being coated but instead "bonded"), they are nevertheless still present. Hence the poly(meth)acrylate resins which have become established in the sealing of polystyrene or of PVC, such as
20 DEGALAN® P 24 or DEGALAN® N 80, cannot be used, since with these products it is not possible to achieve sealed-seam strength.

DEGALAN® P24 is a copolymer of methyl methacrylate and
25 butyl methacrylate and is marketed by Röhm GmbH & Co. KG. DEGALAN® N80 is a methyl acrylate-ethyl methacrylate copolymer and is likewise marketed by Röhm GmbH & Co. KG.

30 With DEGALAN® PM 555 (manufacturer: Röhm GmbH & Co. KG), a special, solvent-borne binder system based on polyolefin/poly(meth)acrylate in butyl acetate/methyl ethyl ketone, success has been achieved in developing a binder possessing outstanding suitability for the
35 sealing of aluminium foils to polypropylene. However, with DEGALAN® PM 555, furthermore, it is also possible to implement equally effective sealing to polystyrene and PVC. To achieve aluminium adhesion, DEGALAN® PM 555 has to be primed with PVC copolymers such as VINYLITE®

VMCH, for example, but in appropriate combination it can also be used as a one-coat system. VINYLITE[®] VMCH is an acid-containing PVC polymer (sales: Union Carbide).

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DEGALAN[®] 4150-E (manufacturer: Röhm GmbH & Co. KG), finally, was developed in order to improve the known heat-seal composition for aluminium foils and plastic films so that instead of the hitherto customary solvent-borne binder system, comprising butyl acetate and methyl ethyl ketone, it is possible to use a solvent system which is not listed in the HAPs list (Hazardous Air Pollutants).

15 **Prior art**

The place of receptacles and containers made of wood or of inorganic materials such as metal, glass and ceramics in foodstuffs packaging has at the present time been taken in many instances by plastic products. The requirements are in any case particularly exacting when the receptacles or containers are used to accommodate or preserve foodstuffs.

One decisive aspect in foodstuff preservation, whether it be dehydration, freezing or sterilization, is the complete prevention of microbial growth. In many cases this means that the containers must be given a gastight seal. Moreover, in the case of plastic containers, account must be taken of mechanical stability, control of water content, and the effects of atmosphere and light. (Cf. Ullmann's Encyclopedia of Industrial Chemistry 25th Ed. Vol. A11, 523 - 560; 583 - 618 VCH 1988; this reference also deals with the applicable standards.)

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In foodstuffs technology, particularly in the case of dairy products such as yoghurt cups, closure of plastic receptacles is widely achieved by using aluminium lids

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coated with a sealing lacquer.

Aluminium lids of this kind constitute in many cases a three-layer laminate whose outer layer is frequently composed of (biaxially oriented) polyethylene terephthalate (O-PET), biaxially oriented polypropylene (OPP) or polyamide (O-PA), or of cellulose. The heat-sealable inner layer, in contrast, is commonly composed of polyethylene, ethylene copolymers, or polypropylene (US-A 4 753 708; G. Stehle, Neue Verpackung 9/91, pp. 94 - 101). Against the use of aluminium for the application, for example, of foodstuffs packaging, however, economic and environmental grounds are more and more entering the debate, particularly the high level of energy involved in producing the aluminium. Consequently aluminium is also being replaced by the use of plastic films provided with a sealable lacquer. The sealing lacquer is adapted to the plastic used. As a relatively inexpensive material for sealable films, with good mechanical strength and good barrier properties, a quite considerable part is played by unplasticized PVC, the sealing lacquer layer employed commonly being an acrylic resin, whose adhesive strength and melting point can be advantageously modified by means of additives. A growing importance is being accorded to PET films. PET films are normally sealed using polyvinyl acetate or polyethylene/polyvinyl acetate, or polyester lacquers. These systems, however, have been developed specifically for the sealing of PET base materials and are of only limited suitability for sealing aluminium foils or aluminium coatings on composite films. To date there has been no disclosure of a heat-seal binder which when formulated appropriately is suitable for sealing both aluminium foils and PET films or films/foil coatings.

DE-A 35 31 036 describes plastic films which can be produced by coextrusion and are composed of a sealable

layer of high-impact polystyrene, a block copolymer and a lubricant.

EP-A 0 406 681, however, rightly points out the
5 problems associated with the use of heat-sealable plastic films rather than aluminium foil. A noticeable - and restrictive - manifestation as a general rule is the substantially reduced processing latitude. The processing range which results is
10 predominantly very narrow, 10 - 20 C, and must be observed with a fair degree of consistency in order to guarantee undisrupted production and flawless use of the sealed pack. In the case of filling installations which have a plurality of cavities for the purpose of
15 filling cups simultaneously, this condition is not always met. The object which EP-A 0 406 681 set itself was to improve the polystyrene-based films preparable by the coextrusion process in accordance with DE-A 35 31 036 in such a way, among others, that the
20 processing latitude and processing reliability are increased. Furthermore, the intention was to ensure flawless production even on filling installations featuring a plurality of filling cavities. In practice this comes down to the use of relatively high sealing
25 temperatures, with corresponding requirements imposed on the quality of the plastic films. According to EP-A 0 406 681 these requirements were met by a sealable plastic film produced by the coextrusion process or by lamination and comprising two layers, A
30 and C, and, optionally, a layer B, and also, optionally, one layer each of an adhesion promoter D for joining each pair of layers A, optionally B and C, composed of 1% to 50% of a layer of a heat-sealable, high-impact polystyrene A, up to 95% of a base layer B
35 and 1 to 99% of a high-melting plastic layer C, the total of the thicknesses or of the weight of A and optionally B and C being 100 in each case.

DE 101 50 898 (Röhm) relates to a heat-sealing system comprising an olefin polymer or olefin copolymer A, a methacrylate copolymer B and a graft polymer AX comprising the above components, and also a solvent or
5 solvent mixture. The heat-sealing system is notable for high thermal stability and short seal times.

WO 2005/037548 (Showa Denko Plastic Products Co., Ltd.) describes an easy-to-open closure for packaging.

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The sealing film is composed of two plies:

- a sealing layer
- and
- 15 - a primer.

The sealing layer is composed of

- A) a copolymer of ethylene and alkyl (meth)acrylates
20 or vinyl ester and

- B) a polyester resin.

The primer is composed of component A of the sealing
25 layer and a polyolefin (C).

The disadvantages of the system lie in the need for priming and in the fact that in the primer the relatively high-value alkyl (meth)acrylates are used
30 which are unable to contribute anything to the properties of the sealing layer.

Furthermore, the polyester resin (B) possesses a very high melting point, which of course complicates the
35 production of the layers.

A further disadvantage is that the good properties of the coating are achieved by double application of the

organic material.

Object

- 5 The object was to develop a heat-seal lacquer binder which, in a formulation with polyesters, is suitable for closing not only aluminium foils but also PET films and not only aluminium-coated but also PET-coated films to all of the cup materials used, such as polystyrene,
10 polypropylene or polyvinyl chloride. In addition, the seal ought to possess seal and barrier properties, with respect to both foil/film materials, which are at least comparable with, if not better than, those of the established heat-seal systems (e.g. Degalan® PM 555)
15 with respect to aluminium foils. Furthermore, in a formulation without added polyester, this binder ought to exhibit an at least equivalent sealing performance to aluminium foils as that of established systems.
- 20 A further object was to achieve high adhesive strength, even at relatively high temperatures, immediately after sealing (high thermal stability), since in this way short cycle times are achieved for the sealing of the film and for the foodstuffs packager.
- 25 Yet a further object is to improve the known heat-seal composition for aluminium foils and plastic films in such a way that in place of the hitherto customary solvent-borne binder system, comprising butyl acetate and methyl ethyl ketone, it is possible to use a
30 solvent system which is not listed on the HAPs list. A further aim is to achieve sufficient strengths of the closure within the usual cycle times employed for the heat sealing of aluminium foils or plastic films. By
35 readjusting the solvent system the intention, finally, is to achieve an additional improvement in seal properties with respect both to aluminium and - in appropriate formulation - to PET films.

The object, furthermore, was to develop a heat-seal composition which remains stable in/to the printing operation which follows the application of the heat-seal composition. Prior-art binders can lead to deposits on the transport rolls of the printing machine after short printing times, so that reliable transport of the film to be printed is no longer possible. The printing machine has to be halted, partly dismantled, and cleaned. The change-over and cleaning times required for this are in some cases out of proportion to the running time of the machines. The binders of the invention allow trouble-free printing over a long period.

Achievement

The object is achieved by means of a film-forming dispersion of at least three different polymer types, A, B, AB, C and D or D_A,

- polymer type A being an olefin copolymer or olefin polymer,
 - polymer type B being a methacrylate polymer, and
 - 25 ➤ polymer type AB being a graft polymer of polymer type A and polymer type B, and
 - polymer type C being a polyester, and
 - optionally one or more further polymer types D or D_A, which are different from the polymer types A -
 - 30 C, and
- a solvent or solvent system L.

Polymer type A

35 The olefin polymers and copolymers for use in accordance with the invention, corresponding to A, are known per se. They are primarily polymers synthesized from ethylene, propylene butylene and/or other

α -olefins having 5-20 carbon atoms, of a kind which have already been recommended for use as heat-sealable compositions. The molecular weight is situated in general at 10 000 to 300 000, preferably between 50 000 and 150 000. Olefin copolymers of the type to be employed are described for example in German Laid-Open Specifications DE-A 16 44 941, DE-A 17 69 834, DE-A 1939 037, DE-A 19 63 039 and DE-A 20 59 981.

As an olefin polymer it is possible for example to use Buna[®] 6170 (manufacturer: Lanxess AG).

Particularly suitable for use are ethylene-propylene copolymers; likewise possible are terpolymers involving addition of the known ter components, such as ethylidene-norbornene (cf. Macromolecular Reviews, Vol. 10 1975), although account must also be taken of their tendency towards crosslinking in the course of the aging process. The distribution may be largely random, though it is also possible with advantage to employ sequenced polymers containing ethylene blocks. The ethylene-propylene monomer ratio here is variable within certain limits, which can be set at about 95% for ethylene and about 95% for propylene, as an upper limit.

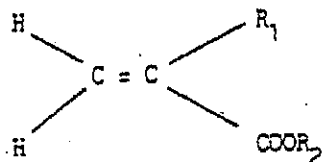
The amounts in which polymer type A is used in the mixture of the invention are between 15% and 65% by weight, preferably between 20% and 60% by weight and, with very particular preference, between 25% and 55% by weight.

Polymer type B

is composed, as defined, of polyacrylate and/or polymethacrylate sequences. Taken per se, in the form of a corresponding homopolymer or copolymer, for example, these are soluble in the solvent system L. The

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polymer B is generally synthesized to an extent or more than 50% by weight, preferably 80% to 100% by weight, of monomers of the Formula I



I

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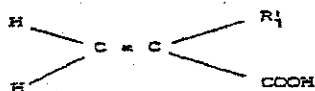
in which R_1 is hydrogen or methyl and R_2 is an alkyl radical, an aliphatic or aromatic radical having 1 to 30 carbon atoms, preferably 1 to 20 carbon atoms.

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In the polymer B the following may additionally be present:

Monomers of the Formula II

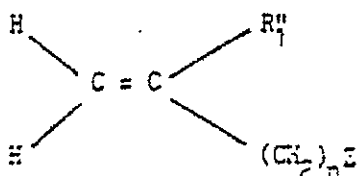
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II

in which R'_1 is hydrogen or methyl, and/or polymerizable acid anhydrides, and/or monomers of the Formula III

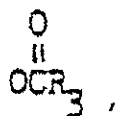
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III

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in which R''_1 is hydrogen or methyl and Z is an optionally alkyl-substituted phenyl radical, a radical $-\text{COR}_3$, a radical

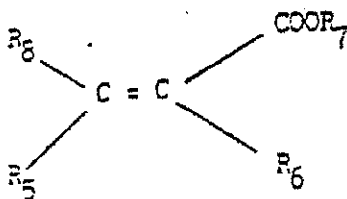


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a radical $-\text{OR}_4$ or a chlorine atom, and in which R_3 and R_4 are each an optionally branched alkyl radical having 1 to 20 carbon atoms or a phenyl radical, and n is 0 or

10 1,

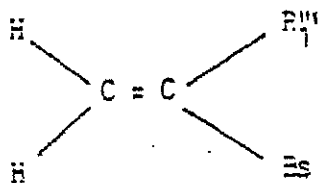
and/or monomers of the Formula IV



IV

15 in which R_5 and R_6 are hydrogen or a radical $-\text{COOR}'_7$, R_6 being hydrogen or a radical $-\text{CH}_2\text{COOR}''_7$, with the proviso that the compound of the Formula IV must contain two carboxyl-containing groups, and in which R_7 , R'_7 , and R''_7 are each hydrogen or an optionally

20 branched alkyl radical having 1 to 20 carbon atoms, or phenyl. The polymer B may optionally further include fractions of the monomers of the Formula V



V

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in which R'''_1 possesses the definition of R_1 and Bs is a nitrogen-containing functional radical such as a -CN group, a group $-\text{CONR}_9\text{R}_{10}$ in which R_9 and R_{10} independently of one another are each hydrogen or a
 5 alkyl radical having 1 to 20 carbon atoms, or in which R_9 and R_{10} , with incorporation of the nitrogen, form a heterocyclic 5- or 6-membered ring, or in which Bs is an (inert) heterocyclic radical, in particular a pyridine, pyrrolidine, imidazole, carbazole or lactam
 10 radical or alkylated derivatives thereof, or Bs possesses the definition $-\text{CH}_2\text{OH}$, or in which Bs possesses the definition



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in which Q is an optionally alkyl-substituted alkylene radical having 2 to 8 carbon atoms and R_{11} is -OH, is -OR''', or is a radical $-\text{NR}'_9\text{R}'_{10}$, with R'''_7 , R'_9 and R'_{10} possessing the same definitions as R_7 , R_8 and R_9 :
 20 for example, together with the nitrogen atom, optionally with incorporation of a further heteroatom, forming a five- to six-membered heterocyclic ring.

Examples of the monomers of the Formula I include
 25 methyl methacrylate, ethyl methacrylate, propyl methacrylate, butyl methacrylate and isobutyl methacrylate. The monomers of the Formula I are also referred to as standard (meth)acrylates.

30 Examples of the monomers of the Formula II include acrylic acid or methacrylic acid.

Examples of monomers of the Formulae III and IV include, in particular, styrene, α -methylstyrene, vinyl
 35 chloride, vinyl acetate, vinyl stearate, vinyl methyl ketone, vinyl isobutyl ether, allyl acetate, allyl

chloride, allyl isobutyl ether, allyl methyl ketone, dibutyl maleate, dilauryl maleate and dibutyl itaconate. The fraction of the monomers of the Formula II-V as a proportion of the polymer B is in general
5 between 0% and 50% by weight, preferably 0% to 20% by weight (relative to the monomers of the polymer B). The fraction of the monomers of the Formulae II and/or V as a proportion of the polymer B will generally not exceed 20% by weight, and in general is 0% to 10% by weight,
10 preferably 0% to 5% by weight.

Specifically, the polymer B, in terms of fraction and composition, is advantageously chosen with regard to the desired technical function. The fraction of the
15 polar monomers of the Formulae II and V will in general not exceed 20% by weight, and is preferably 0% to 10% by weight (relative to the monomers of the polymer B), more preferably 0% to 5% by weight. Monomers of the Formula V include C- and N-vinylpyridine and also
20 vinylpyrrolidine, vinylpyrrolidone, vinylcarbazole, vinylimidazole and the alkyl derivatives thereof, especially the N-vinyl compounds, and also the hydroxyl and dialkylaminoalkyl esters of acrylic and/or of methacrylic acid, especially dimethylaminoethyl
25 (meth)acrylate, dimethylaminopropyl (meth)acrylate and hydroxyethyl (meth)acrylate.

The amounts in which polymer type B is used in the mixture of the invention are between 15% and 65% by
30 weight, preferably between 20% and 60% by weight and, with very particular preference, between 25% and 55% by weight.

In the heat-seal system there is additionally a polymer
35 AB, which represents a polyolefin grafted by B.

Polymer type AB

Preparation of graft polymers AB

The graft polymer AB is generally prepared by preparing a dispersion of component A, with the aid of an appropriate emulsifier, and grafting component B onto this component A under the reaction conditions suitable for the purpose. The emulsifier may possess a structure similar to that of the system AB. The processes for preparing suitable emulsifiers of type AB are known per se. For example, it is possible to proceed in accordance with the method of transfer grafting (cf. also Houben-Weyl, Methoden der Org. Chemie, Vol. 1411, p. 114, H. A. J. Battaerd, G. W. Tregear, Polymer Reviews, Vol. 16, Interscience (1967)).

This is done by preparing a 10% - 50%, preferably 20% - 30% strength by weight solution of an OCP in a suitable solvent, inert under polymerization conditions, which normally possesses a boiling point above the process temperature. Examples of suitable solvents include butyl acetate, aliphatic cycloaliphatic and aromatic hydrocarbons. These OCP solutions are admixed with the monomers of the Formula I and/or, where appropriate, the other monomers II-III in the proportions indicated, and polymerization is carried out with the addition of one or more, preferably peroxidic, free-radical initiators at temperatures of 60 degrees C - 50 degrees C over a time, commonly, of 4 - 8 hours. The aim is for maximum conversion. Preference is given to using peresters such as tert-butyl peroctoate. The initiator concentration is guided by the number of grafting sites desired and by the desired molecular weight of the segment B. In general the initiator concentration is between 0.2% and 3% by weight, relative to the polymer.

Optionally it is possible to use regulators as well in order to set the desired molecular weight of the

segments B. Examples of suitable regulators include sulphur regulators, especially mercapto-containing regulators, an example being dodecyl mercaptan. The regulator concentrations are generally 0.1% to 1.0% by weight, relative to the overall polymer. Another method of preparing the graft polymers AB is the hydroperoxidation of an OCP as the first step. The chain-sited hydroperoxide groups formed in this way are able in a subsequent stage to initiate the graft polymerization of the vinyl monomers. (Cf. H. A. J. Battaerd, G. W. Tregear, Polymer Reviews loc. cit.)

One process for preparing suitable block polymers consists, for example, in anionic polymerization. In this case one possible procedure, for example, is to polymerize isoprene and/or butadiene with a suitable anionic initiator (e.g. organometallic compound) and to react the "living" anionic chain ends with, for example, alkyl methacrylate and/or styrene. The polymers thus prepared are subsequently hydrogenated under conditions in which the functional groups present are not attacked. For details of the preparation reference may be made to the relevant literature, e.g. Houben-Weyl, Methoden der Org. Chemie, 14/1, p. fff.; Block Copolymers, 5 D. C. Allport, W. H. Janes, Appl. Sci. Publishers Ltd., London, 1973; Graft Copolymers, H. A. J. Battaerd, G. W. Tregear, Polymer Reviews Vol. 16 (1967); Block and Graft Polymers, W. J. Burlant, A. S. Hoffmann, Reinhold Publishers Corp., New York, 1960.

The amounts in which polymer type AB is used in the mixture of the invention are between 15% and 70% by weight, preferably between 20% and 65% by weight and, with very particular preference, between 25% and 55% by weight.

Polymer type C

As polymer type C use is made in accordance with the invention of a copolyester which is marketed under the brand name DYNAPOL[®] by Degussa AG. In addition it is also possible to use low molecular mass polyester types, and also polyesters having linear or branched alkyl groups; the polyesters may also be substituted by hydroxyl-containing alkyl groups. As polymer type C it is preferred to use the type DYNAPOL[®] LS 415, which is a saturated, linear, highly elasticized copolyester of very high molecular mass.

The properties of polymer type C in the case of DYNAPOL[®] LS 415 are as follows:

Parameter	Value	Unit	Measurement method
Viscosity number	75 - 85	cm ³ /g	DIN 53 728
Acid number	< 3	mg/KOH/g	DIN EN ISO 2 114
Hydroxyl number	5	mg/KOH/g	DIN 53 240
Molar mass	25 000	g/mol	calculated
Glass transition temperature	12	°C	DSC
Softening temperature	110	°C	ISO 4 625
Density at 25°C	1.14	g/cm ³	ISO 1 183

The viscosity number was measured on a solution of 0.5 g of polyester and 100 g of a mixture of phenol/o-dichlorobenzene (50/50 w/w).

The properties of polymer type C in the case of DYNAPOL[®] H 826-05/A are as follows:

Parameter	Value	Unit	Measurement method
Viscosity (23 degrees C)	3.0 - 5.5	Pa s	DIN EN ISO 3 219
Acid number (solvent-free)	< 3	mg/KOH/g	DIN EN ISO 2 114
Hydroxyl number (solvent-free)	18 - 24	mg/KOH/g	DIN 53 240
Molar mass	6000	g/mol	calculated
Glass transition temperature (solvent-free)	30	°C	DSC
Non-volatiles content	55 ± 1	% by weight	DIN EN ISO 3 251
Density (20°C) (solution as supplied)	1.08	g/cm ³	DIN 51 757
Colour number (Gardner)	> 3		DIN ISO 4 630
Glass transition temperature (solvent-free)	30	°C	DSC
Flash point	58	°C	DIN 53 213

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This is a saturated, linear, hydroxyl-containing polyester resin of medium molecular mass. It is marketed in a solvent mixture of 80% by weight naphtha 150 and 20% by weight naphtha 200, with a content of 55% by weight.

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The properties of polymer type C in the case of DYNAPOL® L 208 are as follows:

Parameter	Value	Unit	Measurement method
Viscosity number	65 - 75	cm ³ /g	DIN 53 728
Acid number	4 - 10	mg/KOH/g	DIN EN ISO 2 114
Hydroxyl number	3 - 9	mg/KOH/g	DIN 53 240
Molar mass	20 000	g/mol	calculated
Glass transition temperature	65	°C	DSC
Softening temperature	155	°C	ISO 4 625
Density at 20°C	1.25	g/cm ³	ISO 1 183

- 5 The viscosity number was measured on a solution of 0.5 g of polyester and 100 g of a mixture of phenol/o-dichlorobenzene (50/50 w/w). This is a saturated, branched polyester of high molecular mass.

The properties of polymer type C in the case of DYNAPOL[®] H 820-20 are as follows:

Parameter	Value	Unit	Measurement method
Viscosity (23 degrees C)	1.5 - 2.5	Pa s	DIN EN ISO 3 219
Acid number (solvent-free)	< 3	mg/KOH/g	DIN EN ISO 2 114
Hydroxyl number (solvent-free)	15 - 25	mg/KOH/g	DIN 53 240
Molar mass	5000	g/mol	calculated
Glass transition temperature (solvent-free)	60	°C	DSC
Non-volatiles content	40 ± 1	% by weight	DIN EN ISO 3 251
Density (20°C) (solution as supplied)	1.07	g/cm ³	ISO 1 183
Colour number (Gardner)	> 3		DIN ISO 4 630
Flash point	82	°C	DIN 53 213

- 5 This is a saturated, linear, hydroxyl-containing polyester resin of medium molecular mass. It is marketed in a solution in naphtha 200 (40% by weight).

10 The amounts in which polymer type C are used in the mixture of the invention are between 5% and 50% by weight, preferably between 5% and 30% by weight and, with very particular preference, between 10% and 25% by weight, the weight figures relating to the polymer without the possible solvent fraction.

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Polymer type D or D_A

To improve the aluminium adhesion a formulation with polymer type D is an option: priming the film or adding
20 0% to 10% by weight, better still 0% to 5% by weight, of polyvinyl chloride to the lacquer formulation allows

an improvement to be observed in the Al adhesion. Use is made of, for example, VINYLITE[®] VMCH (marketed by Union Carbide).

- 5 Polymer type D_A can be added optionally in order to reduce possible abrasion during processing. Use may be made of a polyamide of the VESTOSINT[®] 2159 (Degussa AG) type, for example. This can be added in a formulating fraction of between 0% and 10% by weight, better still
10 between 0% and 5% by weight.

From preliminary experiments it is known that PET adhesion with a binder system as described above, comprising components A, B, AB and optionally D and/or
15 D_A, in a solvent system comprising n-butyl acetate and ethyl methyl ketone (in analogy, for example, to Degalan[®] PM 555), in a formulation with the polymer component C, exists only with unsatisfactory sealing strengths, irrespective of the concentration of
20 component C. Changing the solvent system over to an inventive composition described below, in contrast, leads to very good heat-seal properties in appropriate formulation with polymer component C.

25 Solvent or solvent system L

In addition to the polymer types A and B, the graft polymer AB, the polymer type C and optionally the polymer type D or D_A, there is also a solvent system in
30 the heat-seal system of the invention.

The solvents to be used for the solvent system L are to be selected such that they meet the requirements of coating technology for plastics and metals. The
35 solvents to be employed - in a mixture if appropriate - are to be inert and, as a whole, unobjectionable, the boiling point as far as possible not exceeding 105 C at 760 torr.

A suitable solvent is a mixture of esters of aliphatic carboxylic acids with aliphatic alcohols and aliphatic hydrocarbons.

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A suitable aliphatic carboxylic acid is acetic acid, propynoic acid or butyric acid.

Suitable aliphatic alcohols are ethanol, propanol,
10 isopropanol, n-butanol, 2-butanol, 2-methyl-1-propanol
or 2-methyl-2-propanol.

Suitable aliphatic hydrocarbons are pentane, hexane,
heptane, octane, isooctane, nonane, decane, undecane or
15 dodecane.

The aliphatic hydrocarbons may also be present in the
form of their isomer mixtures and in mixtures with one
another. Cycloalkanes, including substituted
20 cycloalkanes, can be used as well.

When selecting L, attention should be paid to the
compatibility between polymer components A and B. In
the case of incompatibility between A and B, L may
25 represent an equally good solvent for both components,
in which case the dispersion forms as a result of the
incompatibility.

In the case of compatibility between A and B, L must be
30 chosen such that it represents a better solvent system
for B than for A at a given temperature; the dispersion
then forms on the basis of the different dissolution
quality. In accordance with the invention the solvent L
chosen is preferably a solvent such that the olefin
35 copolymers can be swollen in the temperature range
between 40°C and 150°C to 5% to 300% by weight ("degree
of swelling").

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The degree of swelling is defined as follows: an OCP film 1 mm thick, 2 cm long, 0.5 cm wide and of known weight is immersed in the solvent at a defined temperature - at 90°C in the exemplified case - and stored isothermally for 24 hours, then removed from this solution using tweezers, freed from adhering swelling agent by means of a paper filter, and then immediately weighed. The measure defined for the swelling is the weight increase in per cent, relative to the initial weight. The swelling measurement ought to be carried out at the temperature at which the concentrated OCP dispersion is to be produced. In accordance with the invention the swelling at this temperature ought to be 5% to 300% by weight. A precondition for the applicability of the criterion is that the maximum swelling of the OCP is attained under the conditions indicated above.

Mixtures of the above-described solvents are also suitable for the carrier system.

The fraction of the solvent or solvent system L as a proportion of the inventively concentrated polymer dispersions can be, for example, 80% by weight, in a particularly favourable case to down to 20% by weight, preferably below 70% by weight, in practice usually 55% to 40% by weight.

Production of the heat-seal system

Formulation with polymer types A - D

Alternative A:

Using an appropriate emulsifier, a dispersion or, in the case of a suitable match of polymer compatibility, a homogeneous solution of components A and C in the solvent system L is prepared, as elucidated above, and under appropriate reaction conditions component B is

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grafted onto component A. The proportions of A and B are generally in weight ratios of approximately 1:5 to approximately 5:1. The weight ratio of A to C is generally between approximately 1:2 and approximately 10:1, preferably between approximately 1:1 and approximately 5:1.

Alternative B:

10 Using an appropriate emulsifier, a dispersion or, in the case of a suitable match of polymer compatibility, a homogeneous solution of component A in the solvent system L is prepared, as elucidated above, and under appropriate reaction conditions component B is grafted
15 onto component A. The proportions of A and B are generally in weight ratios of approximately 1:5 to approximately 5:1. The dispersion thus prepared is subsequently admixed with polymer component C. The weight ratio of A to C is generally between
20 approximately 1:2 and approximately 10:1 - better still between approximately 1:1 and approximately 5:1.

The amount of the overall polymer, based on the overall dispersion, independently of the preparation process,
25 is at least 10% by weight, the aim in practice being for 40% to 80% by weight, normally 45% to 60% by weight.

The process of the invention produces heat-sealable
30 coating compositions in dispersion, which possess sufficient stability for the processing operation. The stability of the dispersions amounts to at least several days, normally several weeks to months.

35 The heat-seal systems of the invention are notable for good adhesive strengths and for thermal stability. They can be used for sealing packaging, especially foodstuffs packaging.

Examples

Film/foil material used

5.

Soft aluminium foil with a thickness of 40 µm
Polypropylene film, extruded from JE 6100 pellets
(Shell), thickness 0.2 mm
Polyester film: Hostaphan® WDW 50

10

Heat-seal solution formula

Prior to processing, DEGALAN® PM 555 or 4150 E must be
agitated thoroughly, since on prolonged storage there
15 is a possibility of phase separation.

The heat-seal binders have a solids content in their
as-supplied form (manufacturer: Röhm GmbH & Co. KG) of
approximately 45% by weight and for further processing
20 are diluted with methyl ethyl ketone (MEK) to
approximately 30% by weight. For promoting adhesion on
the aluminium foil, PVC copolymers have proved to be
appropriate. They are either applied as a primer or
used in combination with the heat-seal binders. In our
25 investigations, combination took place with Vinylite®
VMCH 95/5. For the coating of the polyester film,
DEGALAN® was combined 90/10 and 80/20 with DYNAPOL®.

Laboratory application of heat-seal solution

30

The primer was applied as a 10% by weight solution of
Vinylite® VMCH in ethyl acetate to the aluminium foil
(10 cm × 20 cm) using a No. 2 K-Handcoater. This
corresponds to a dry film thickness of 0.5 µm - 1.5 µm.

35

The DEGALAN® heat-seal solution was applied using the
No. 4 K-Handcoater. Dry film thicknesses of 7 µm - 9 µm
were obtained.

Laboratory drying of coated films/foils

The primed films/foils were flashed off briefly
5 (5 - 10 minutes) and then dried at 180°C in a forced-air oven for 15 seconds.

Heat sealing and determination of sealed-seam strength

10 The sealing operations were carried out using the heat sealer (HSG/ET) from Brugger.

Sealing conditions:

15	Temperature:	200°C
	Pressure:	6 bar
	Time:	1 sec.
	Seal area:	10 × 10 mm

20 The sealed-seam strength was determined by cutting samples into strips 15 mm wide and subjecting them to tension at a speed of 100 mm/min. using the tensile testing machine from Instron, model No. 1195, or Zwick, model No. 1454. Care was taken to ensure that, during
25 the peel test, the parts of the foil/film already separated from one another formed an angle of 90°C with the as yet unstressed remainder.

PP cups were sealed using a VL-1600 laboratory cup
30 sealer from BHS.

Results of experiments

Comparison of solvent systems

Heat-seal properties

Designation		Solvent L	HSF (N/15 mm)	
			Al/PP	PET/PP
Example 1		Propyl acetate - isooctane - ethyl acetate	12	7.8
Example 2		ditto		7.0
Comparative 1		Butyl acetate - methyl ethyl ketone	approx. 10	1.1
Comparative 2		ditto		3.2

Composition of the polymer
of Examples 1 - 2:

EPDM/PMMA/PMBA/polyester
42.9/18.55/18.55/20% by
weight

Composition of solvent L:

Propyl acetate: 54% by
weight
iso-octane: 9.5% by weight
Ethyl acetate: 36.5% by
weight

Composition of the polymer
of Comparative: 1 - 2:

EPDM/PMMA/PBMA/polyester
44/18/18/20% by weight

Composition of solvent L:

Butyl acetate: 70% by weight
Methyl ethyl ketone: 30% by
weight

- 5 Both for Example 1 and for Comparative 1, the fraction of polymer is approximately 45% by weight and the fraction of solvent is approximately 55% by weight.

- 10 EPDM = Buna EP 6170
PMMA = polymethyl methacrylate
PBMA = polybutyl methacrylate
Polyester = Dynapol LH 826

CLAIMS

1. Heat-sealable coating system suitable for sealing different kinds of substrates and composed of a film-forming dispersion of at least three different polymer types, A, B, AB, C and optionally the polymer types D or D_A, and an organic solvent system L,

characterized in that

10

- polymer type A is an olefin polymer or olefin copolymer,
- polymer type B is a (meth)acrylate copolymer containing standard (meth)acrylates and in total up to 15% by weight of methacrylic acid and/or acrylic acid and/or another polymerizable acid, relative to polymer type B,
- polymer type AB is a graft polymer of polymer type A and polymer type B, and
- 20 ➤ polymer type C is a polyester or a polyester mixture;
- optionally there is also a mixture of polymer type D or of different polymer types D_A, and

25 in that the amount of polymer type A is between 15% and 65% by weight,

in that the amount of polymer type B is between 15% and 65% by weight,

30 in that the amount of polymer type AB is between 15% and 70% by weight,

in that the amount of polymer type C is between 5% and 50% by weight,

in that the amount of polymer type D is between 0% and 10% by weight,

35 in that the amount of polymer type D_A is between 0% and 10% by weight, based on the total mass of polymer types A - D,

and in that the mass ratio of the total of polymer

- 27 -

types A - D to the mass of the solvent or solvent mixture L is between 5:1 and 1:5.

2. Heat-sealable coating system suitable for sealing
5 different kinds of substrates, according to Claim 1,

characterized in that

10 the amount of polymer type A is between 20% and 60% by weight,
the amount of polymer type B is between 20% and 60% by weight,
the amount of polymer type AB is between 20% and 65% by weight,
15 the amount of polymer type C is between 5% and 30% by weight,
the amount of polymer type D is between 0% and 5% by weight,
the amount of polymer type D_A is between 0% and 5% by
20 weight,
based on the total mass of polymer types A - D and D_A.

3. Heat-sealable coating system suitable for sealing
different kinds of substrates, according to Claim 1,
25

characterized in that

the amount of polymer type A is between 25% and 55% by weight,
30 the amount of polymer type B is between 25% and 55% by weight,
the amount of polymer type AB is between 25% and 55% by weight,
the amount of polymer type C is between 10% and 25% by
35 weight,
the amount of polymer type D is between 0% and 5% by weight,
the amount of polymer type D_A is between 0% and 5% by

weight,

based on the total mass of polymer types A - D and D_A.

4. Heat-sealing system according to Claim 1 to 3,

5

characterized in that

the solvent system is a solvent mixture comprising
components

10

L1.) esters of aliphatic carboxylic acids with
aliphatic alcohols and

L2.) aliphatic hydrocarbons,

15 the weight ratio of L1.) to L2.) being between 1 and
200 and the boiling point of the solvent system under
standard conditions being not more than 105 degrees
Celsius.

20 5. Solvent system according to Claim 4,

characterized in that

it is composed of a mixture of aliphatic hydrocarbons
25 having 5 - 12 carbon atoms in the chain, and the chain
may also be branched.

6. Solvent system according to Claim 4,

30 characterized in that

it is composed of a mixture of esters of aliphatic
carboxylic acids having 2 - 4 carbon atoms in the
chain, with aliphatic alcohols having 2 - 4 carbon
35 atoms in the chain, and the chains may also be
branched.

7. Solvent system according to Claim 4,

characterized in that

5 it is composed of a mixture of aliphatic hydrocarbons
having 5 - 12 carbon atoms in the chain, and the chain
may also be branched, and of a mixture of esters of
aliphatic carboxylic acids having 2 - 4 carbon atoms in
the chain with aliphatic alcohols having 2 - 4 carbon
atoms in the chain, and the chain may also be branched.

10

8. Solvent system according to Claim 4,

characterized in that

15 the volume ratio between esters and aliphatic
hydrocarbons is between 1 and 200.

9. Solvent system according to Claim 4,

20 characterized in that

the boiling temperature of the solvent system is
between 70 degrees Celsius and 105 degrees Celsius.

25 10. Solvent system according to Claim 4,

characterized in that

30 the boiling temperature of the solvent system is
between 85 degrees Celsius and 100 degrees Celsius.

11. Method of sealing heat-sealable substrates,

characterized in that

35

a coating system according to any one of the preceding
claims is used.

- 30 -

12. Method according to Claim 11,

characterized in that

5 an aluminium foil or a polyester film is coated with the coating system and sealed to a plastic selected from the group of the plastics polypropylene, polyethylene, polystyrene, polyester or polyvinyl chloride.

10

13. Solvent system according to Claim 10,

characterized in that

15 it is composed of a mixture of propyl acetate (40% to 70% by volume), ethyl acetate (24% to 45% by volume) and isooctane (5% to 15% by volume).

14. Packaging system,

20

characterized in that

it is sealed with a heat-sealing system according to Claim 1.

25

15. Food packaging system,

characterized in that

30 it is sealed with a heat-sealing system according to Claim 1.