Polym. Bull. (2011) 66:491–501
DOI 10.1007/s00289-010-0289-7

ORIGINAL PAPER

Modified and functionalized maleimide copolymers for paper coatings

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Received: 28 January 2010 / Revised: 23 April 2010 / Accepted: 2 May 2010 /

Abstract New functional aqueous dispersions of poly(styrene-co-maleimide) (SMI) and poly(octadecene-co-maleimide) (OMI) have been prepared by grafting different functional additives into the copolymers side chains to explore the possibility of tailoring the properties of these auxiliary pigments for applications in paper coating formulations. Modifications were carried out by reacting the corresponding maleic anhydride (MA) copolymer with ammonia and small amounts of various functional primary amines such as 4-amino-2,2,6,6,-tetramethylpiperidine (TAD), L(+)-aspartic acid (ASP), 2,2,2-trifluoroethylamine (TFEA) and 2,2,3,3,4,4,4-heptafluorobutylamine (HFBA) to investigate opportunities to fine-tune weathering stability (TAD), adhesion properties (ASP) or hydrophobicity (TFEA, HFBA) of the coated paper board. The thermal imidazation reaction of MA groups was carried out at 155 °C for 6 h in organic solvent-free process using high shear forces. The obtained functional nanoparticles varied in size from 50 to 217 nm and exhibited high glass transition temperatures (T_g) ranging from 150 to 180 °C. Paper board coating trials are also reported in this article.

Keywords Polyimides · Pigments · Paper coating · Surface modification

Introduction

Poly(styrene-co-maleic anhydride) (SMA) and poly(octadecene-*alt*-maleic anhydride) (OMA) have a similar structure except for the phenyl moiety in SMA that has been replaced by a 16 carbon long aliphatic side chain in OMA. Both are inexpensive commercially available polymers with many desirable properties such

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as miscibility with other styrenic copolymers and high thermal stability which is proportional to the maleic anhydride (MA) content [1–7]. A well-known route to further increase the thermal stability of an organic substance is to, e.g. synthesize an imide (MI) from a cyclic anhydride with an amine [8, 9]. Consequently, the use of analogous reaction on copolymers, i.e. imidization of the MA unit with ammonia, makes it possible to obtain MI copolymers with significantly increased glass transition temperatures and mechanical properties [8–12]. Whereas, the synthetic strategy to partially replace ammonia with a functional primary amine offers a convenient way to control and fine-tune the properties of the produced polymer for various applications [1, 13–18]. Vermeesch et al. [14] reported chemical modification of SMA with primary *N*-alkylamines by reactive extrusion, Becker et al. [15] with amino alcohols in the melt, Pu et al. [16] with 2-amino-benzimidazole and Wang et al. [1] with a fluorescent dye, 4-amino-*N*-(2,4-dimethylphenyl)-1,8-naphthalimide. Moreover, Tavana and Appelhans et al. [17, 18] modified OMI by introducing perfluorinated side chains to obtain a highly hydrophobic surface.

Organic polymers, both natural and synthetic, are prone to thermal and photo-oxidation at nearly all stages of their life cycle. The degradation process will ultimately lead to polymer failure due to undesirable changes in the physical and or/visual properties of the polymer, e.g. to embrittlement and discoloration. The weathering resistance of polymers can be improved by addition of hindered amine light stabilizers (HALS) that are prepared from 4-amino-2,2,6,6,-tetramethylpiperidine (also known as triacetonediamine, TAD) and related compounds. Numerous mechanisms of stabilization have been proposed, and consensus has been reached that the nitroxyl radicals $>NO^{\bullet}$, derived from the HALS explain the UV-stabilizing efficiency of the parent hindered amine [19]. Conventional HALS stabilizers are low-molecular weight compounds that are known to migrate out of the polymer matrix—this can be overcome by using higher molecular weight HALS derivatives and polymeric HALS or reactive HALS that can be chemically tethered to the polymer backbone [20–23]. Signh et al. [24] have prepared TAD-*graft*-SMA to make polymeric HALS for stabilizing high impact polystyrene (HIPS). However, to the best our knowledge there are no reports on HALS functionalized SMI copolymers.

L(+)-Aspartic acid (ASP) and its homopolymer are widely used in biomedical applications [25, 26]. Hydrophilic segments can be introduced to non-polar polymers by incorporation of aspartic acid moieties whereby surface and bulk properties of the host material can be controlled and fine-tuned for various applications. Willett et al. [27] studied adhesion of amino acids including aspartic acid to different inorganic surfaces making it possible to regulate adhesive properties of the surfaces. Shogren et al. [28] have prepared low molecular weight copolymers of aspartic acid by reactive extrusion, and Kakuchi et al. [29] have successfully reacted aspartic acid with phthalic anhydride.

By incorporating fluorochemicals, it is possible to make the surface hydrophobic. Fluorochemicals affect surface tension and thus the wetting properties of the material [17, 18, 30, 31]. Improved barrier properties can also be achieved via treatment with fluorochemicals [32]. There are several reports on the successful surface engineering of maleimide copolymers through attachment of fluorinated

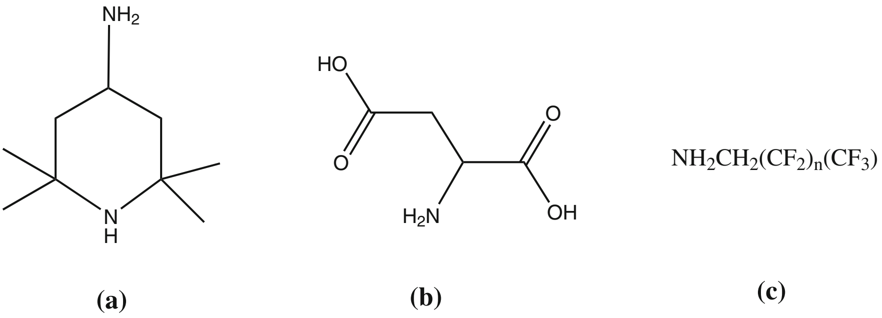

Fig. 1 Functional amines used for modification of SMI and OMI: **a** 4-amino-2,2,6,6,-tetramethylpiperidine (TAD), **b** L(+)-aspartic acid (ASP) and **c** 2,2,2-trifluoroethylamine (TFEA, $n = 0$) and 2,2,3,3,4,4,4-heptafluorobuthylamine (HFBA, $n = 2$)

side chains to create the desired surface characteristic features for a host of different applications [17, 18, 30].

In this study, we investigated the possibility and effect of incorporating small amounts of functional side chains to poly(styrene-co-maleimide) and poly(octadecene-co-maleimide) nanoparticles through imidization with 4-amino-2,2,6,6,-tetramethylpiperidine, L(+)-aspartic acid and two fluoroamine derivatives (see Fig. 1). The resulting auxiliary nano-particle pigments with added functional molecular groups have been applied to the surface of paper sheets to hopefully improve their printing characteristics.

Materials and methods

Materials

Poly(styrene-co-maleic anhydride) (SMA, M_w 80,000 g/mol, 26 mol.% maleic anhydride) was received from NV Topchim SA. Poly(octadecene-*alt*-maleic anhydride) (OMA, M_n 30–50,000 g/mol, 50 mol.% maleic anhydride), ammonia (NH_3 25% aq. solution), 4-amino-2,2,6,6-tetramethylpiperidine (TAD, 98%) and 2,2,2-trifluoroethylamine (TFEA, 99.5%) were purchased from Sigma-Aldrich and 2,2,3,3,4,4,4-heptafluorobutylamine (HFBA, 97%) and L(+)-aspartic acid (ASP, 98+%) from Acros, Fair Lawn, NJ, USA. All coating chemicals and additives were kindly provided by BASF (former Ciba Speciality Chemicals). All chemicals were used as received.

The synthesis of modified maleimide copolymers

The polymerization of aqueous dispersion of these *N*-substituted maleimide copolymers (Fig. 2) was conducted in a 1-L Büchi autoclave with an anchor stirrer. SMA/OMA (1 *n* maleic anhydride unit), 0.855 *n* ammonia, 0.095 *n* modifying compound (TAD/ASP/TFEA/HFBA) and water were added into the

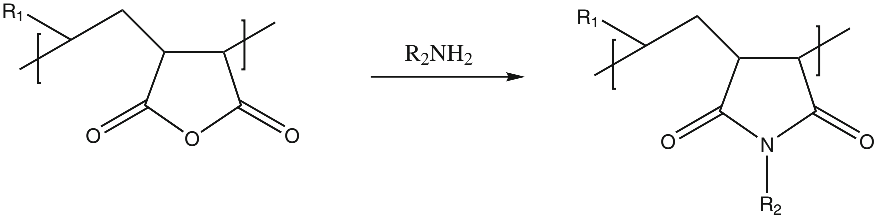

Fig. 2 Modification reaction of maleimide copolymers where R_1 is phenyl (SMI) or hexadecyl (OMI) and R_2 is –H (SMI/OMI) and 4-piperidinyl (TAD), 2-butanedioic acid (ASP), 2,2,2-trifluoroethyl (TFEA) or 2,2,3,3,4,4,4-heptafluorobuthyl (HFBA)

reaction vessel in one batch to produce dispersion with a solid content of 20%. Agitation (200 rpm) was started, and the reaction vessel was heated to 155 °C with Lauda oil bath and the reaction was allowed to continue for 6 h. Finally, the reaction was cooled down to room temperature, and the obtained polymer dispersion was analyzed and used as such.

Coating colour preparation

Coating colour was prepared by using 100 pph kaolin (Nugloss or Capim NP) as primary pigment or by replacing part of the primary pigment with 10 pph synthetic pigment SMI/OMI, latex (LTX 316, 10 pph), carboxyl methyl cellulose (CMC, FinnFix 30, 0.5 pph), calcium stearate, hardener and optical brightening agent (Tinopal ABP-Z, 0.6 pph). The components were mixed, and pH was regulated by sodium hydroxide (NaOH) to 8.5.

Laboratory trials

Coating colours were coated on one side at 10 g/m^2 on precoated paper board (Stromsdal). The experiment was carried out using a laboratory scale Helicoater. Coated paper boards were then calendered at a laboratory calender (once against both cylinders at 90 °C with no additional pressure). All the samples were conditioned for 24 h at 23 °C and 50% relative humidity before analysis.

Measurements

Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR, Sensir Durascope Perkin-Elmer FTIR 1000) was used for collecting infrared spectra. Particle sizes of the nanopoarticle dispersions were measured by light scattering on Malvern ZetaSizer Nano ZS and glass transition temperatures by differential scanning calorimeter (DSC; TA Instruments DSC Q1000) using both traditional and modulated techniques. Dynamic surface tension of the liquid was measured using maximum bubble pressure method on SensaDyne 9000 Max bubbel tensiometer which was calibrated with water and ethanol. Surface tension was registered when the value had stabilized. Paper analyses were performed according to valid TAPPI and/or SCAN paper testing standards. Accelerated ageing test was performed in a Weiss climate cabinet at 80 °C and 65% relative humidity (RH) for 72 h, and the received CIE Lab colour values were recorded on Elrepho SF 450.

Results and discussion

Dispersions

Recently, we have investigated in detail the influence of various reaction parameters such as agitation rate and stirrer type, reaction temperature, time and molar ratio of ammonia on the thermal imidization of poly(styrene-co-maleic anhydride) (SMA) and poly(octadecene-co-maleic anhydride) OMA [8]. The results show that reaction temperature of 150 °C, reaction time of 5 h, shearing power of anchor type mixer of 200 rpm and ammonia to anhydride molar ratio of 0.9 are adequate reaction conditions for complete imidization of MA groups and for obtaining an odourless product with no residual ammonia. Under these reaction conditions in both cases, hard nanoparticles were formed due to strong hydrogen bonding between maleimide units in these particles. Initially, we wanted to explore whether under similar reaction conditions one could partially replace ammonia by a small amount of various functional primary amines without the loss of dispersion stability and particle size. One of the most important criteria for possible synthetic coating pigment is that their particle size is not in the submicron scale but truly under 150 nm, i.e. with a higher specific surface area. In practise, co-pigment cannot be added into coating formulations above 5–10 wt%. In order to obtain an improved effect on paper coatings in terms of runnability, gloss, brightness and printability, one should increase the number of added particles by constant added volume. This can best be achieved by reducing the particle size preferably to 100 nm or less, as shown in Fig. 3.

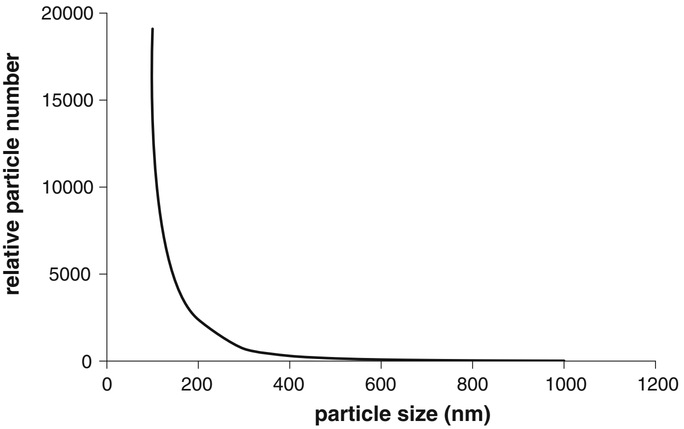

Fig. 3 Particle size versus relative particle number

The results from synthesizes of modified SMI dispersions are shown in Table 1. All of the modified maleimide nanoparticles formed stable dispersions. Thus, the introduction of functional primary amines did not disturb or prevent the formation of nanoparticles. Also the content of hydrogen bonding in the nano-sized SMIs was sufficient to ensure high dispersion stability. The modified SMI particles were all monodispersed in size and had nano-particle sizes ranged from 50 to 217 nm, whereas their glass transition temperatures (T_g) varied between 150 and 180 °C. In general, modifications raised glass transition temperatures, and functionalized SMI dispersions had higher T_g values and smaller particle sizes than the corresponding OMI copolymers.

Modified maleimide copolymers were characterized by attenuated total reflection infrared spectroscopy (ATR-FTIR). The spectra for chemically modified OMI and unmodified OMI are depicted in Fig. 4. In all modifications, the characteristic imide

Table 1 Characteristics of modified maleimide copolymers

Sample	Modifier (wt%)	Particle size (nm)	T_g (°C)
SMI	–	114	165
SMI-TAD	3.5	71	180
SMI-ASP	3	50	176
SMI-TFEA	2.2	87	174
SMI-HFBA	4.42	117	169
OMI	–	69	150
OMI-TAD	1.91	180	148
OMI-ASP	1.63	217	164
OMI-HFBA	2.42	182	NA

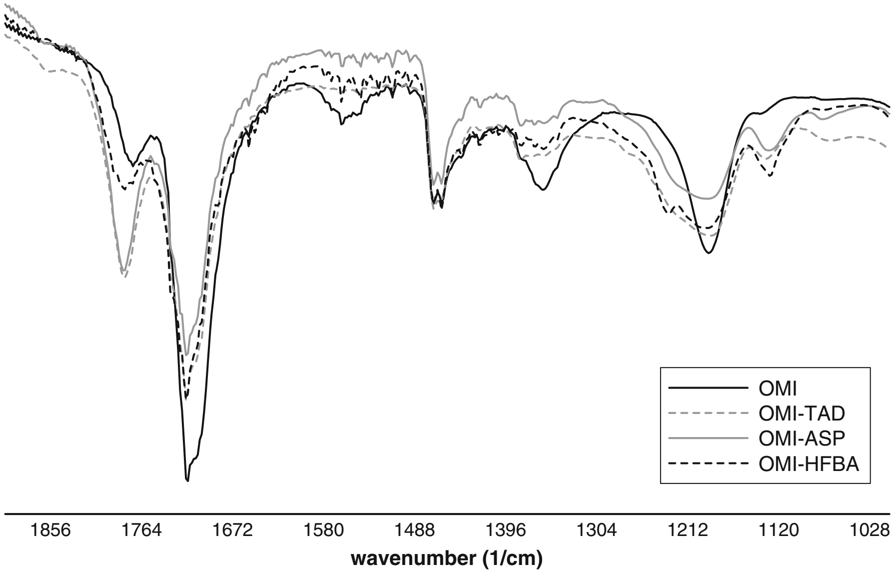

Fig. 4 IR spectra of *N*-substituted poly(octadecene-co-maleimide)

absorption bands at 1,715 cm^{-1} (C=O symmetric stretching), 1,780 cm^{-1} (C=O asymmetric stretching) and 1,380 cm^{-1} (C–N stretching) are present, whereas the typical absorption bands for anhydride unit at 1,850 cm^{-1} (C=O stretching) are either missing or are very small [31]. Thus, the ATR-FTIR data clearly suggest that the imidization reaction has been successful. The presence of perfluoroalkyl in OMI-HFBA is indicated by the new peak at 1,220 cm^{-1} (CF_3 stretching). In the OMI-ASP case, the fairly strong absorption at 1,770 cm^{-1} can be attributed to succinic acid end groups.

Maleimide copolymer dispersions modified with HFBA were further analyzed using a SensaDyne 9000 Max bubble tensiometer. The unmodified SMI and HFBA modified SMI possessed surface tension values of 84 and 74 mN/m, respectively. In the case of OMI, only a very small decrease in surface tension could be recorded, i.e. 70 mN/m for OMI versus 69 mN/m for OMI-HFBA. As expected, fluorination decreased surface tensions even though the decrease was very small in the case of OMI. This can partially be attributed to the relatively low concentration of HFBA units in OMI.

Paper board coating

The state-of-art applications of nanotechnology in the paper industry are mainly focused on the paper formation production process to eliminate chemical additives controlling dewatering with nanoparticles of bentonite and colloidal silica. Thus, the long-term goal of the industry is to achieve better dewatering performance by replacing these additives with nanotechnology. Whereas in the paper coating sector so far, to the best of our knowledge, mainly unbound nanoparticles of poly(styrene-co-acrylate) polymers have been applied to improve coating quality. Our interest in researching the functional SMI and OMI nanoparticles has been to explore whether the modified functional nanoparticles could successfully be applied to the surface of paper sheets to improve their performance and printing characteristics. As of today, offset printing can be considered to be the dominantly used method of printing with a market share of over 40%. Offset lithography relies on a very simple principle that hydrophobic ink and hydrophilic fountain solution are incompatible and do not mix. Thus, the tacky ink adheres to the image area whereas the fountain solution to the non-image area. First, a combination of ink and fountain solution is distributed on a printing cylinder on which the image is created and then it is transferred to a rubber blanket cylinder that prints the image on paper. In offset printing, the ink splits in the nip whereby a strong perpendicular tack force is imposed on the surface of paper. These forces cause the paper to climb up the cylinder rolls and may lead to picking/blistering (delamination). Therefore, a certain z-strength or pick-strength of coating is required for successful printing [33, 34]. In addition, we envisioned that by appropriate selection of the functional primary amine one should be able to predictably design and fine-tune surface properties of paper coatings. Table 2 lists the obtained data from coating colour and paper analysis.

All of the prepared synthetic maleimide copolymer pigments increased water retention of the coating colour in comparison to the reference containing only primary pigments. Dewatering affects not only process runnability and drying of the

Table 2 Coating colour and paper analysis

Sample	GWR[c] (g/m^2)	Br 100[d] (Pas)	Gloss (%)	PPS 10[e] (µm)	Brightness D65 (%)	K&N ink absorption (%)	IGT pick (m/s)
Ref 1[a]	56	213	53	1.7	89.2	5.9	1.3
SMI[a]	24	172	35	2.0	91.0	1.8	1.8
SMI-TAD[a]	36	170	39	2.2	90.4	1.7	1.8
SMI-ASP[a]	21	172	44	2.2	90.3	1.8	1.3
SMI-TFEA[a]	33	163	41	2.2	90.0	1.3	1.8
Ref 2[b]	101	185	43	1.9	87.0	7.4	1.7
OMI[b]	55	154	39	2.4	89.3	5.5	4.0
OMI-TAD[b]	88	244	29	2.0	88.6	2.4	2.6
OMI-ASP[b]	98	247	49	1.3	84.8	2.0	2.1
OMI-HFBA[b]	96	225	38	2.0	88.4	0.7	2.9

[a] Primary pigment Capim NP

[b] Primary pigment Nugloss

[c] ÅA GWR water retention

[d] Brookfield 100 viscosity

[e] Parker Print Surf surface roughness

coating layer but also physical properties of coated paper and the final print quality. Dewatering property is a function of particle interactions, base paper properties and coating conditions. Some coating techniques, such as applicator roll coating, benefit from greater dewatering whereas in blade coating this is generally viewed as a problem. Excess dewatering causes a dramatic increase in solid content and thereby decreases the runnability of the process [35, 36]. In addition, the measured viscosities for the produced nanoparticles were in a good range for optimal flow properties. As a consequence of this, the runnability of all prepared coating formulations was very good. Surface roughness of the coated and calendered paper boards were somewhat increased and gloss was diminished. On the other hand, brightness, K&N ink absorption and IGT pick were all slightly improved. The IGT test measures the ability of a coated surface to resist picking or blistering during offset printing. The measurement is conducted with IGT laboratory printing device where a paper or board sample is printed with oil of known viscosity at accelerating printing speed (e.g. according to standard SCAN-P 63). The speed at which picking is observed is taken as a measure for picking resistance. As could be anticipated, maleimide nanopigments modified by ASP had the lowest IGT pick values which could be either a result of decreased coating cohesion or strong ink-coating adhesion. Furthermore, K&N ink absorption was lowest for the hydrophobic-fluorinated maleimide nanopigments. This result is also inline with earlier findings which have shown that papers treated with fluorochemicals have high repellent characteristics towards other organic compounds such as grease/oil and exhibit improved barrier properties [32].

Three days of accelerated ageing at 105 °C (ISO 5630-1) correspond to 25 years of natural ageing. However, this method does not take into account the presence of environmental humidity which has led to the development of a moist method (ISO

5630-3) [37]. The impact of TAD on weathering was tested according to this moist method in a Weiss climate cabinet at 80 °C and 65% RH for 72 h. The decrease in CIE whiteness was almost constant despite the presence of TAD: whiteness drop in SMI and SMI-TAD were 21.6 and 21.5 units, respectively. Thus, the desired improvement in weathering stability was not fully achieved by this modification. This lack of improved light stability in terms of CIE whiteness can be at least partially attributed to the fact that the stabilizer may not be present in a sufficient concentration in the right phase and up to a reasonable depth. In addition, it has been shown that polymeric 2,2,6,6-tetramethylpiperidine additives show low activity in polymers that have a high degree of hydrogen bonding. This is due to the fact that >N–H group forms intramolecular hydrogen bonds that prevent their conversion to the active nitroxyl radicals (>N–O) which in turn are necessary for stabilization of polymers [24].

Conclusions

We have successfully modified both poly(styrene-co-maleimide) and poly(octadecene-*alt*-maleimide) copolymers by adding small amounts of functional groups such as 4-amino-2,2,6,6,-tetramethylpiperidine, L(+)-aspartic acid and fluorinated compounds through a thermal imidization reaction in aqueous media to give organic nanoparticles. The pre-studies clearly indicate the feasibility of this nano-scale engineering technique. The modified copolymers have been applied in paper coating as auxiliary pigments and we have been able to show that fluorinated groups and aspartic acid affect surface properties. In general, all the coating formulations increased water retention of coating colour. Whereas, in the case of tethering 2,2,6,6-tetramethylpiperidine moieties onto the nano-pigments, no significant improvement of light stability could be observed. Indeed, full effect of the modifiers was not yet achieved, and thus further work is needed to establish which amount of functional groups are necessary for optimal performance in various paper coating formulations.

Acknowledgements We would like to thank the Finnish Funding Agency for Technology and Innovation (TEKES), Center of Excellence for Functional Materials (FunMat), BASF (former Ciba Speciality Chemicals) and NV Topchim SA for scientific advice and funding of the project. We would also like to thank Johan Norrgård for laboratory assistance at Laboratory of Polymer Technology, Åbo Akademi University.

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