



Attributes of the latex emulsion processing and its role in morphology and performance in paints

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ARTICLE INFO

Article history:

Received 11 February 2011

Received in revised form 5 May 2011

Accepted 12 May 2011

Keywords:

Reaction pathway
Acid distribution
Morphology
Scrub
Gloss
Whiteness

ABSTRACT

This article explores the influence of process variation and neutralization of itaconic acid (IA) on polymer properties and paint performance. The study elaborated how the reaction pathway changes the acid distribution in the latexes. The study also investigated the influence on paint performance of degree of neutralization of IA before its incorporation into emulsion. The acid distribution study was done through the conductometric titration method. Latex film properties were studied through differential scanning calorimeter (DSC) and minimum film forming temperature (MFFT). These latexes were formulated in the corresponding paint and study was done using scanning electron microscopy (SEM), which was further correlated with the performance properties of the paint such as scrub, gloss, whiteness and contrast ratio.

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1. Introduction

In recent years, due to environmental regulations, the role of latex in coating formulations is the key of bringing the desired performance. At the same time, there is an increasing demand to develop the latex to be more compatible with paint additives which will provide volatile organic compounds (VOC) compliant coating system.

Thus, in order to meet the ever increasing demand of VOC regulation and to optimize the latex designed there are several reports well recognized in the literature such as incorporation of crosslinkings in the polymer backbone and emulsion process variation and latex particle morphology [1–21].

The most studied route is the incorporation of acid functional polymer as it resulted in the improvement of several significant properties such as colloidal stability, mechanical and freeze–thaw stability, rheology, and adhesion. However, properties of carboxylated lattices are greatly influenced by the method of insertion, nature of the carboxylic monomer and also the distribution of carboxylic acid group in serum, surface or buried in the latex. It has also been observed that the rate of polymerization of bifunctional carboxylic acids such as fumaric [19], maleic acid and more specifically itaconic acid (IA) [1–9] is slower than that of acrylate esters and therefore their presence is likely to reduce the rate of propagation in the aqueous phase. The incorporation of even small amounts

of IA into the copolymer significantly increases the solubility and hence the polarity of the emulsion. These factors are likely to affect the final properties of latexes having methacrylic acid (MAA) and IA [10–12]. Nevertheless, the paint formulator is frequently able to detect even minor changes not anticipated by the latex chemist by the interaction or reaction of obscure ingredients [18]. Perhaps many of these results would be more undesirable, if we could actually measure the impact of these process changes on the latex structure and the effect on the paint performance [2]. Thus it is worth to study the process variation and behavior of acid functional monomers in the latex and its interaction with the paint ingredients. In the present article we have investigated (i) the effect of polymerization processes (ii) effect of neutralization of IA before its incorporation into emulsion polymerization and their performance properties in the paint formulation.

2. Experimental

2.1. Materials

Methyl methacrylate (MMA), butyl acrylate (BA) and methacrylic acid (MAA) were of technical grade procured from Evonik Industries, Germany. Itaconic acid (IA) was obtained from Nikhil Adhesives Pvt. Ltd, Mumbai. Fatty alcohol ethoxylate (40 mole EO) from Cognis, whereas, sodium lauryl sulfate (SLS), potassium persulfate, sodium bicarbonate were received from local suppliers, Mumbai. All other materials used for making paints such as calcium carbonate, TiO₂, additives and extenders were of commercial grade in-house raw materials and used without further purification.

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Table 1
Emulsion recipe and characterization.

Ingredients	%
MMA	45–47
BA	
Proprietary monomers	
Acid monomers	0.25 0.50
IA	
MAA	
Surfactants	0.8
SLS	
Fatty alcohol ethoxylate 40 mole (70%)	
Initiator (potassium persulfate)	0.17–0.22
De-ionized water	45–48
Chaser-activator	0.08–0.1
TBHP and SFS	
Emulsion characterization	
% Solid	49–51
pH of emulsion	3.5–4.55
Particle size μm (d_{50})	0.12–0.13
% free monomers other than acid monomers by GC	0.2–0.3
% Free IA by GC	Not detected
% Free MAA by GC	0.005

2.2. Latex preparation

The recipe for latex preparation used in this study shown in Table 1. Two reaction pathways were adopted for processing the emulsion; Process I, in which monomers were pre-emulsified using surfactant water and monomers. The mixture was added at regular intervals in the reactor along with potassium persulfate as an initiator, whereas in Process II, surfactant, IA and monomers were added separately in the reactor without pre-emulsification of the monomers. In order to further study the effect of degree of neutralization of IA, samples of latexes were synthesized by neutralizing IA at 10, 25 and 50% level using sodium bicarbonate (NaHCO_3) before they were incorporated into emulsion polymerization.

The emulsion copolymerization of MMA, BA, IA and MAA was carried out at 78–80 °C in a glass reactor using potassium persulfate as an initiator. Polymerization was carried out by batch process under monomer starved condition for 3.5 h. After completion of monomer feed, reaction was held at 82–84 °C for 30 min and chaser-activator tert-butyl hydro peroxide (TBHP) and sodium formaldehyde sulfoxide (SFS) were subsequently added to complete the polymerization. Mixture of anionic and non-ionic surfactants was used to stabilize the emulsion.

All the experiments were carried out by keeping the composition of surfactants, monomers, and initiators same throughout the study. Table 1 depicts the final emulsion characterization including % free monomers as well.

3. Characterization

3.1. Particle size analysis

Particle size of the synthesized emulsions was measured on Zeta Potential Analyzer. Zetatrac Model NPA152. Microtrac Inc. A Unit of Nikkiso (USA, PA).

3.2. Determination of acid distribution in the emulsion [2]

An accurately weighed sample of latex was diluted to 125 mL with de-ionized water in a suitable beaker and pH of the emulsion was adjusted to 10–11 with dilute NaOH. The sample was then titrated against standard H_2SO_4 in 0.5 mL increments. The allowed time between two increments was 1 min. Conductance was stud-

ied on Conductometer as a function of volume of standard acid consumed until pH of about 2.4 was reached.

3.3. Scanning electron microscopy (SEM)

Paint film was drawn on release paper and before coalescence film (18–20 μm DFT) was flash dried at 80 °C. Electron micrographs were obtained on JEOL Scanning Electron Microscope (SEM) model JSM-5400 (Japan, Tokyo). Sample was mounted on brass stub and coated by gold sputtering method. Samples were scanned at different magnification.

3.4. Differential scanning calorimetry (DSC)

DSC study was done on Q 10 model with Refrigerated Cooling System from TA instruments (USA). Samples were heated at the rate of 10 °C/min under N_2 atmosphere.

3.5. Minimum film forming temperature (MFFT)

Latex film of 50–75 μm was drawn on polyester sheet using bar coater and tested on MFFT bar. Temperature gradient was kept between –5 and 40 °C.

3.6. Scrub resistance [1]

Internal standard based on combination of DIN 53778 Part 2 and ASTM D 2486 was used to determine the wet scrub of the paint by using Washability and Scrub Resistance Tester, Erichson Model 494 (Germany, Iserbach). The method uses the wild boar hair brush and stainless steel brush holder having standardised mass 250 g and aqueous dispersion of silica as an abrasive medium.

This test method is generally used to assess the film integrity and abrasion resistance of paint film. Test samples and control paint samples were drawn side by side on Leneta Scrub test panel using Sheen film applicator. Samples were allowed to cure for 7 days. Then Scrub Panel was oriented over the shim on the glass plate so that coating direction was perpendicular to the direction of the brushes (as per DIN53778). Scrub media (as per ASTM D 2486) was then applied evenly into the brushes which were further attached on a movable arm. The sample was scrubbed to failure and cycle number was recorded.

3.7. Gloss

Gloss measurement was done in accordance with ASTM D 523. Paint film of 150 μm was drawn on Leneta opacity chart and cured for 48 h. Gloss was measured at 20°, 60° and 85° on Erichsen gloss meter model PICOGLOSS 503 (Germany, Iserbach).

3.8. Contrast ratio and whiteness

Contrast ratio was determined in accordance with ISO 6504/3. Paint films were applied on Leneta opacity chart and dried for 48 h. Reflectance measurement was performed on both black and white surface using Gretag Macbeth Color Eye 7000A spectrophotometer (USA, NY) with $d/8^\circ$ geometry.

Whiteness index of the paint coating was determined in accordance with ASTM E313, using R_∞ results derived from the contrast ratio drawdown on Whiteness Hunter 60 colour measurement system.

3.9. Freeze-thaw (F/T) stability of paint

F/T of the paint was determined at –15° to +35°. All paint samples were kept in the freezer at –15 °C for 16 h and then left

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