ISSN: 2616-6445 (Online), ISSN: 2224-5243 (Print)
Комплексное использование минерального сырья №4.2018

UDC 547.022.1
IRSTI 02.04.00
https://doi.org/10.31643/2018/6445.40

Kompleksnoe Ispol′zovanie
Mineral′nogo Syr′a. №4.2018.

EL-SAYED NEGIM[1], L. BEKBAYEVA[2], K. OMURBEKOVA[1]*

*[1] Kazakh-British technical University, Almaty, Kazakhstan, *e-mail: elashmawi5@yahoo.com*
[2] Kazakh national technical University named after K. I. Satpayev, Almaty, Kazakhstan

SYNTHESIS AND CHARACTERIZATION ANTICORROSION EMULSION LATEXES FOR METAL

Received: 29 April 2018 / Peer reviewed 15 September 2018 / Accepted 09 October 2018

Abstract. Copolymer emulsion latexes based on styrene: (St), and 2-ethylhexyl acrylate; (2- EHA), were prepared via emulsion polymerization with different compositions ratios (80:20 %, and 50:50 %). The polymerizations were carried out at 80 °C using ammonium persulfate (APS) as initiator technique system in presence of sodium dodecyl benzene sulfate (DBS) as surfactant. The copolymers lattices were characterized by FT-IR, ^{1}HNMR, TGA, and DSC. Copolymer latexes were high solid content and used as binder in anticorrosive coating for metal. The presence of a new absorption peak in the infrared region of C-H stretching and C-H out of plane bending of polystyrene at 3027 and 696 cm^{-1} and the chemical shift of ^{1}H NMR at 1.5-1.7 ppm due to presence of -CH$_2$- in copolymer shows indicating that the polymerization has taken place. The thermogravimetric analysis (TGA) illustrated higher thermal stability that obtained by decomposition temperature. These copolymers have a single glass transition temperature indicating that these copolymers can form a homogenous phase. However, with increasing the ratio of 2-ethylhexyl acrylate in the copolymer, glass transition temperature increased. The obtained copolymers showed excellent adhesion properties on the metal. Also, the results showed that copolymer latex has good anti-corrosively, UV light stability, and direct application as metal anti-corrosive. However, increasing the ratio of 2-EHA enhanced anticorrosion properties of the metal.

Keywords: copolymer, emulsion, 2-EHA, anticorrosion

Introduction. Metals are used in a wide range of industrial application areas due to high strength and mechanical properties. However, metals corrode when they are exposed to a corrosive environment and lose its protective properties [1]. The anticorrosion coatings are playing a role as a physical barrier layer to separate the surface of metal from corrosive environment. Typical advantages of organic systems: they are mechanically flexible and tough. But the disadvantage consists of having poor abrasion and thermal resistance [2-6]. Anticorrosion coating is fasting-drying and made of pigments suspended in acrylic binder [7-10]. Urban et al prepared acrylic binder based on styrene-acrylic in a reactor. The obtained copolymers were used for the preparation of two component polyurethane coatings for corrosion protection. The results showed that the acrylic copolymers enhanced the protection metal from corrosion [3]. Yixiang et al [6] modified poly (styrene-co-acrylic) with hydroxyl phosphate and exhibits a good performance and corrosion resistance due to the increasing adhesion between coating and metal. In our laboratory [11-17], it is of particular interest to synthesizing different kinds of acrylic binder by different methods including emulsion, solution, grafting and blending polymerization. This article describes the preparation of copolymer binders based on styrene; (St), and 2-ethylhexyl acrylate; (2- EHA), via emulsion polymerization. The obtained copolymers enhanced anticorrosion properties of metal with increasing the ratio of 2-EHA in the copolymer.

Materials and methods. Materials: Details of chemicals used in the study are listed in Table 1.

Table 1 - Chemicals used in this study

Chemical	Shorthand notation	Source
Styrene	St	Sigma, USA
2-Ethylhexyl acrylate	2-EHA	Sigma, USA
Sodium dodecyl benzene sulfate	DBS	Sigma, USA
Ammonium per sulfate	APS	Sigma, USA
Sodium chloride		Sigma, USA
Methanol		Sigma, USA
Acetone		Sigma, USA

Synthesis of the emulsion latexes. Copolymerization of St and 2-EHA were carried out by the technique of semi continuous emulsion polymerization processes with various ratios of monomers. One type of emulsifier, namely, DBS (1 g) was independently used in the emulsion polymerization processes. The emulsifier was dissolved in 50 g of distilled water and charged into a 500 ml three-necked flask. The free radical initiation system of APS (0.2 g) was added to the flask during mechanical stirring at 250 - 300 rpm. The monomers (50 g) (St/2-EHA) monomers were added dropwise (about 12 ml/h) during the stirring process at 65 °C using an automatically controlled water bath under nitrogen atmosphere for a period of 2 h followed by another 1 h at 80 °C. The design of experimental as shown in Table 2.

Table 2 - The experimental design

copoly mer	St	2-EHA	DBS	APS	Water
M1	40 g	10 g	1 g	0.2 g	50 g
M2	25 g	25 g	1 g	0.2 g	50 g

Characterization of emulsion latexes. The prepared copolymers were characterized by Fourier Transform Infra-Red (FT-IR) spectroscopy. Fourier Transform Infra-red (FT-IR) spectroscopy data was used to identify the functional groups. The FT-IR spectrum of prepared copolymers were obtained using (4000 - 400 cm^{-1}) in KBr were recorded using Perkin Elmer 2000 FTIR system, and the Nuclear Magnetic Resonance (NMR). The prepared copolymers were performed by using nuclear magnetic resonance (Bruker Avance 300 and 400, Switzerland). Thermogravimetric Analysis (TGA) and Differential Scanning Calorimeter (DSC) were utilized to study the thermal properties. The per-dried copolymers were tested using Perkin Elmer thermogravimetric Analyzer (TGA/SDTA851^{e}, METTLER TOLEDO, Switzerland). TGA sample weight in the range of 10-15 mg was heated at 20 °C/min room temperature to 895 °C with nitrogen atmosphere. Sample weight in the range of 5- 10 mg was heated at 20 °C/min in the range of 30 – 250 °C with nitrogen atmosphere.

Results and discussions. The structure of prepared copolymers with different composition of styrene, St and 2-ethylhexyl acrylate 2- EHA [M1, (80:20) and M2, (50:50) wt. %, was confirmed by FT- IR and ^{1}H NMR.

FT-IR spectra of the prepared polymers. FT-IR spectra of prepared copolymer with different ratios are shown in Figures 1 and 2. The absorption bands at 3027 and 696 cm^{-1}are characteristics of C-H stretching and C-H out of plane bending of polystyrene. The C-H stretching occurs above 3000 cm^{-1} and is typically exhibited as a multiplicity of weak-to-moderate bands, compared with the aliphatic C-H stretch. The number and positions of the C-H bonds around the ring, which in turn are related to the nature and number of other substituents on the ring, define the structure of the bands. The bands at 1638 and 1493 cm^{-1} results of aryl-substituted C=C bond and the C-C stretching vibration of benzene ring. The other most important set of bands are the aromatic ring vibrations centered around 1600 and 1500 cm^{-1}, which usually appear as a pair of band structures, often with some splitting. The appearance and ratio of these band structures is strongly dependent on the position and nature of substituents on the ring. The absorption bands at 3349 and 1452 cm^{-1}show C-H aromatic stretch and C=C-C aromatic ring stretch of polystyrene. The absorption bands at 2927, 2853, 1727 and 1156 cm^{-1} are the characteristics of stretching methylene C-H, methoxy group O-CH_3, ester C=O and C-O-C alkyl-substituted ether C-O stretch, respectively.

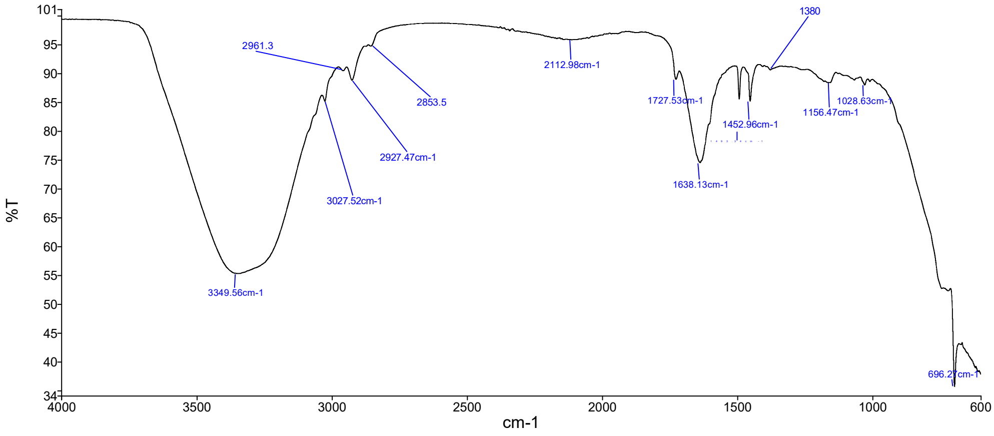


Figure 1- FT-IR spectra of copolymer poly [St-co-2-EHA] with ratios M1

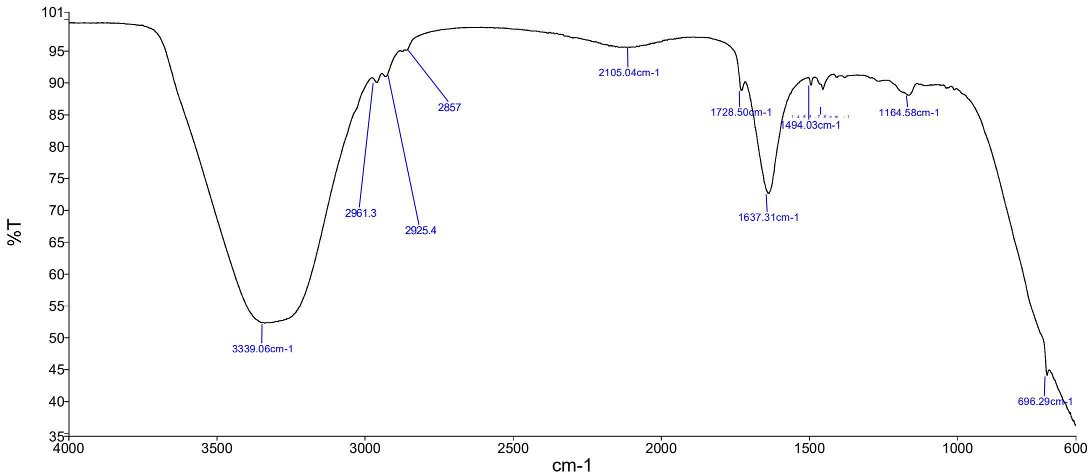


Figure 2- FT-IR spectra of copolymer poly [St-co-2-EHA] with ratios M2

^{1}H NMR. The ^{1}H NMR spectrum provides more evidence for the structure of the prepared copolymers. The typical spectral for ^{1}H NMR is shown in Figures 3 & 4 and Table 2. The chemical shift at δ = 0.88 – 92 ppm is a result of protons in CH_3 groups, δ at range 1.2-1.4 ppm for proton attached to C-2 and δ = 1.5 – 1.7 ppm for proton in C-3 on the ethylhexyl group. The signals at δ 1.98 ppm are assigned to proton attached to C-4. The δ observed at 2.35 ppm is assigned to protons in C-5 and 4.3 – 4.5-ppm protons in C-7 groups attached to the COO side group of 2- EHA. The signals at δ 2.5 ppm are assigned to proton attached to C-6 and 7.2-7.3 ppm to protons in benzene ring. Table 3 shows that there are characteristic peaks of the prepared copolymers. The prepared copolymer is expected to have the following structure according the above characteristics as shown in figure 3.

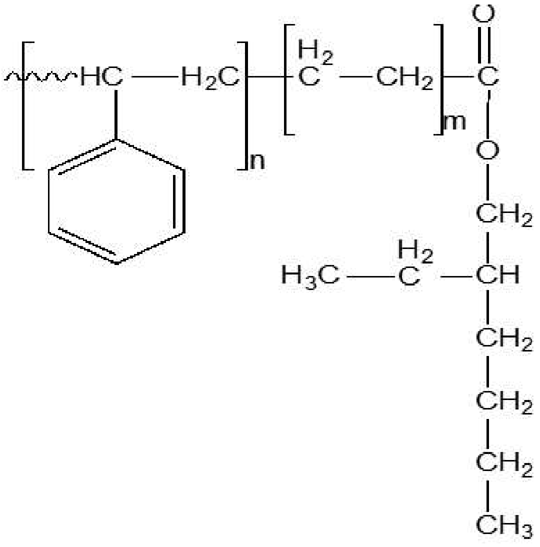
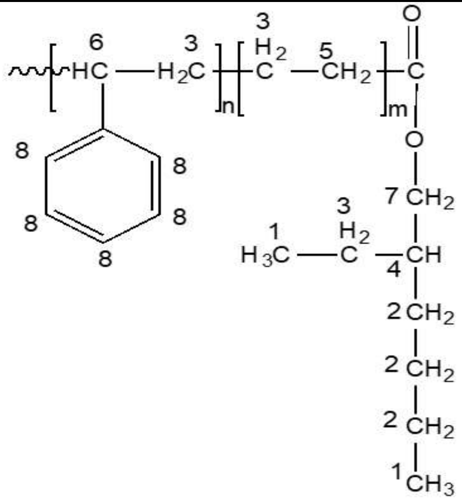


Figure 3- Structure of emulsion latexe

Table 3 - ^{1}H NMR Chemical shifts of the prepared tri-polymers

Resonance Signal (ppm)	Proton	Assignment
7.1-7.3 4.3-4.9 2.5	H [C-8] CH_2 [C-7] CH [C-6]	
2.35	CH_2 [C-5]	
1.9	CH [C-4]	
1.5-1.7	CH_2 [C-3]	
1.2-1.4	CH_2 [C-2]	
0.88-92	CH_3 [C-1]	

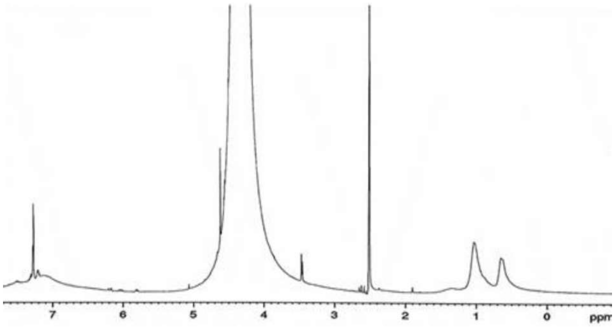


Figure 4 - ^{1}H spectra of prepared copolymer M1 in DMSO-D_6

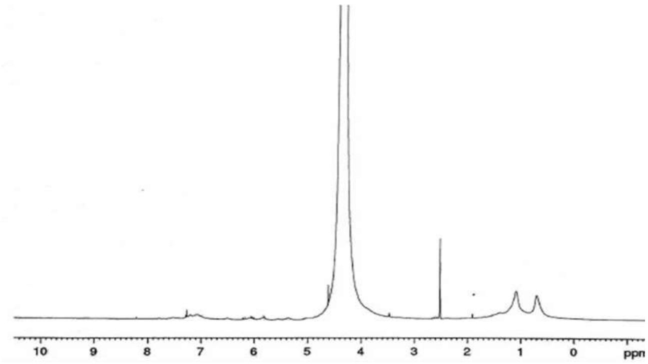


Figure 5 - ^{1}H spectra of prepared copolymer M2 in DMSO-D_6

Differential scanning calorimetry (DSC). Differential scanning calorimetry is a conventional technique to judge the miscibility of a copolymer. A miscible copolymer exhibits one T_g, while an immiscible copolymer displays two T_g's, which correspond to the pure components. Generally, the observation of a single glass transition temperature (T_g) for a copolymer pair, between those of the homopolymers, is regarded as decisive evidence of an unique environment and of polymer miscibility, although different methods of measuring T_g are sensitive to different scales of homogeneity [18]. Differential scanning calorimetry (DSC) was used to assess the extent of copolymerization of St and 2-EHA. Figures 5 & 6 and Table 4 show DSC analysis of obtained copolymer M1 and M2 with different compositions. The copolymer M1 and M2 exhibit two T_g at 61.1 °C, & 154.2 °C and 71.3 °C & 154.3 °C respectively. From Figures 3.6 & 3.7, it is clear that, T_g of the copolymer increases with increasing the ratio of 2-EHA from 20 to 50 %. Furthermore, glass transition of M2 is higher than that of M1, meaning that the M2 is more homogeneous at the molecular scale than the M1.

Table 4 - Thermal properties of copolymers

Copolymers	PDT$_{max}$	Tg^1	Tg^2
M1	420	61.1 ºC	154.2 ºC
M2	410	71.3 ºC	154.3 ºC
Tg: Determined from DSC curves. PDT$_{max}$: Determined from TGA curves.			

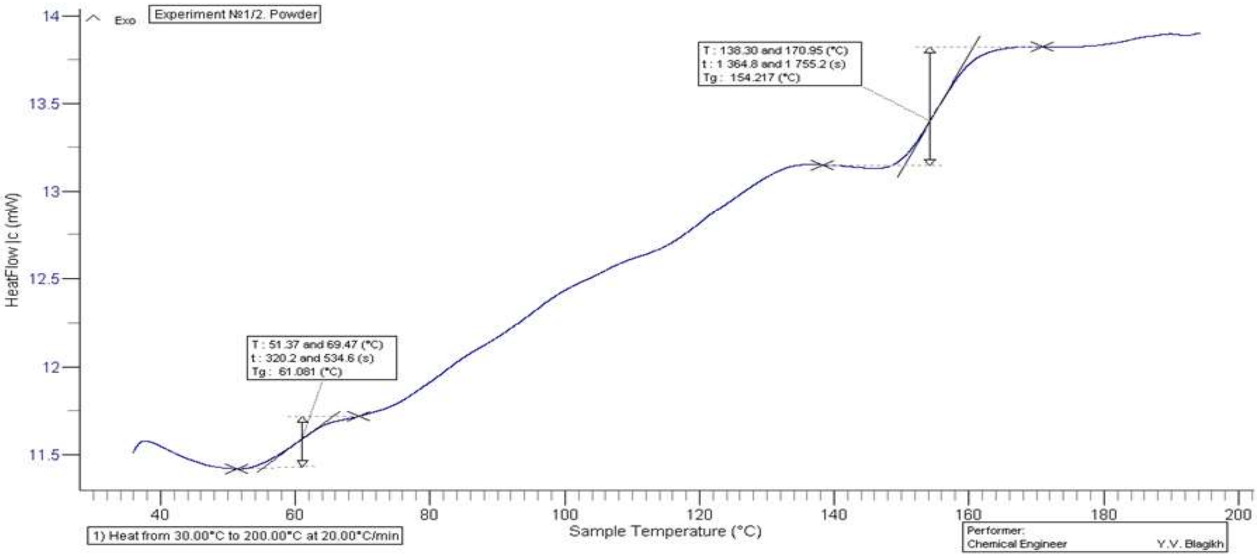


Figure 6 -DSC analysis results of copolymer M1

Thermogravimetric analysis (TGA). Thermogravimetric analysis (TGA) was employed to investigate the thermal stabilities, and the influence of different ratios of St and 2-EHA on the loss of weight in the copolymer. Figures 7 & 8 summarize the TGA results of the copolymers M1 and M2. These TGA's were performed from 0 ℃ to 900 ℃ at 20 ℃/min in air. TGA measurements of copolymers (M1, M2) indicate that thermal degradation temperature is higher than 420 °C and 410 respectively, which is above the highest rheological measurements employed in this study, as shown in the Figures 7 and 8. It is evident that the thermal degradation process for M1 sample proceeds in four steps as shown in Figure 7. The first weight loss occurs between 29 and 150 °C, which corresponds to the removal of water. The second, third and fourth weight loss occurs between 150 and 900 °C correspond to 2-EHA decomposition with loss of CO_2. The maximum polymer degradation temperature (PDTmax) corresponds to the temperature at which the maximum rate of weight loss occurred and appeared at 410 ℃ for copolymers M1 and M2. This behavior is in good agreement with the results obtained by other authors [17-20] for thermal degradation of methylcellulose , according to whom the mechanism of MC degradation includes the parallel processes of dehydration and demethoxylation (—OCH3, —CH2OCH3). In addition, thermal stabilities of copolymer M2 is decomposing in one step.

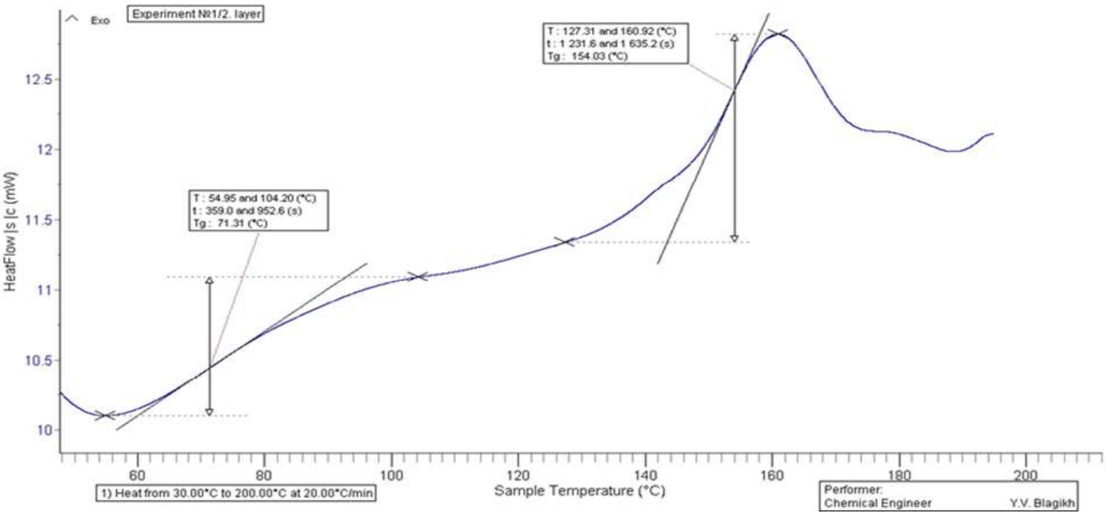


Figure 7 - DSC analysis results of copolymer M2

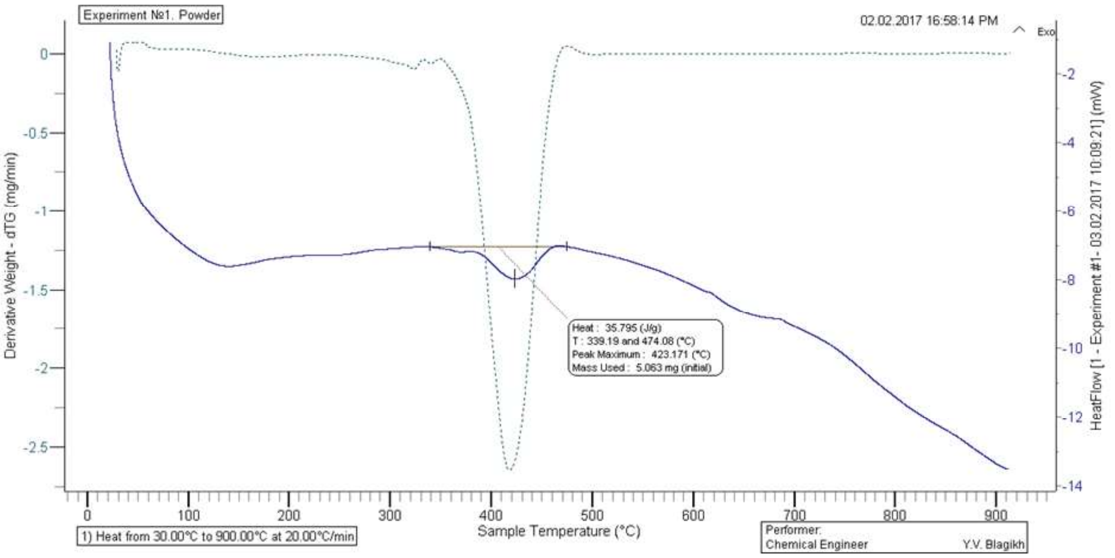


Figure 8 - TGA analysis results of copolymer with ratio M1

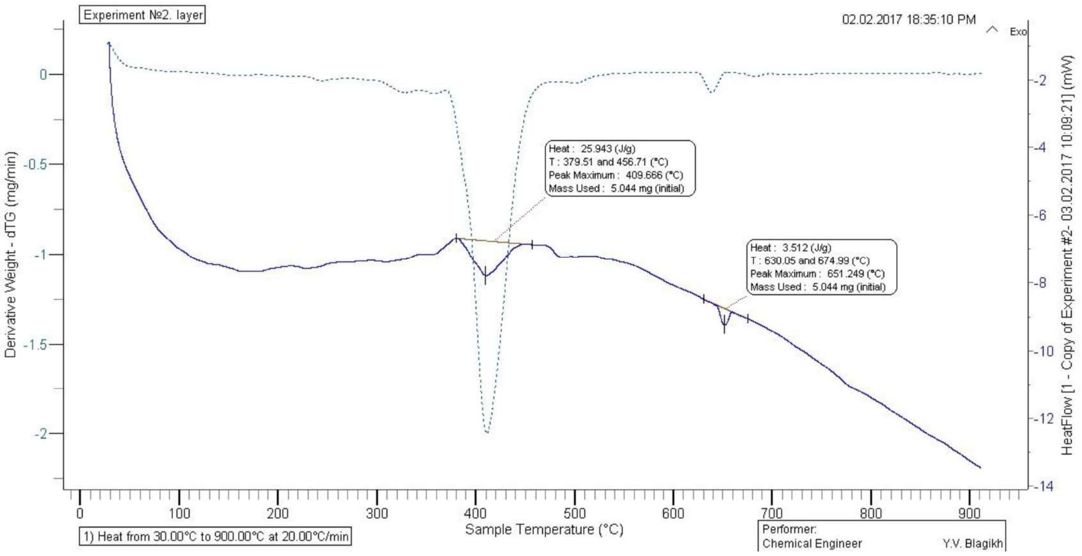


Figure 9 - TGA analysis results of copolymer with ratio M2

Conclusions. In this study, a series of concrete admixtures, copolymers $[(\mathrm{St})_n$- $(2\text{-EHA})_m]$ was synthesized in various compositions of 80 and 50 % w/w respectively, and characterized in relation to FT-IR, ^{1}H NMR, TGA and DSC. The physical-mechanical properties and miscibility behavior of copolymer have been studied by several techniques. Differential scanning calorimetry was the main experimental technique used to study the thermal behavior of the copolymers. Through the measurement of glass transition temperature of copolymer was determined. For copolymers with well-determined glass transition temperature *Tg*, immiscibility of the copolymer was proved by DSC. All the copolymers exhibit two *Tg* and increased with increasing 2-EHA from 20% to 80%. Copolymer samples show good thermal stability. The obtained copolymer latexes enhanced anticorrosion properties of film coating. Acknowledgments. The work was financially supported by Ministry of Science and Education of the Republic of Kazakhstan (program-targeted financing).

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ЭЛЬ-САЙЕД НЕГИМ[1], Л. БЕКБАЕВА[2], Қ. ӨМІРБЕКОВА[1]
*[1] Қазақстан-Британ техникалық университеті Алматы, Қазақстан, *e.mail: elashmawi5@yahoo.com*
[2]Қ. И. Сәтбаев атындағы Қазақ ұлттық техникалық зерттеу университеті, Алматы, Қазақстан

МЕТАЛҒА АРНАЛҒАН КОРОЗЗИЯҒА ТҰРАҚТЫ ЭМУЛЬСИЯЛЫҚ ЛАТЕКСТЕРДІҢ СИНТЕЗІ МЕН СИПАТТМАМАСЫ

Түйіндеме. Әр түрлі құрамы болатын (80:20 % және 50:50 %) стирол негізіндегі (St) сополимерды эмульсиялық латекст және 2 – этилгексилакрилат (2-Эха) эмульсиялық полимерлеу арқылы алынды. Беттік-белсенді заттар сияқты сульфат додецилбензол натрий (DBS) ретінде және персульфат аммонийді бастамаушы ретінде (APS) пайдалану арқылы 80 °C температурасында полимерлеу үрдісі өткізілді. Сополимерлер торлары үшін FT-IR, 1HNMR, TGA және DSC тән. Жаңа шарықтау шегіне сіңіру инфрақызыл облысының C-H созылу және C-H келген жазық иілу полистирол кезінде 3027 және 696 $см^{-1}$ және химиялық ығысу 1H ЯМР кезінде 1.5-1.7 ppm-қатысу -CH_2-сополимере көрсеткендей, полимерлеу үрдісі болатыны анықталды. Термогравиметриялық талдау (ТГА) кезінде ыдырау температурасында жоғарғы термиялық тұрақтылық көрсетілді. Бұл сополимерлерде жеке шыны өткелі температурасы болады, яғни сополимерлер біркелкі телімдерін қалыптастыра алады. Бірақ сополимерде 2-этилгексилакрилат арақатысы ұлғайған сайын шынылау температурасы өсе бастады. Нәтижесінде 2-Эха күшейтілген сайын жабынды қабықшалардың коррозияға тұрақтылығы артатыны көрсетілді. Алынған сополимерлер металда үздік адгезионды қасиеттерін көрсетті. Нәтижелер көрсеткендей, латекс сополимері тотығуға тұрақтылығын және ультракүлгін жарыққа жақсы көрсеткіштері бар екені және жемірілуге қарсы материал ретінде пайдаланылуы мүмкіншіліктері бар екені келтірілді. Сондай-ақ, 2-Эхо ұлғайту арақатынасы күшейткен сайын металдын коррозияға қарсылығы арта бастайды.

Түйін сөздер: сополимері, эмульсия, 2-Эхо, тотығу металлы

ЭЛЬ-САЙЕД НЕГИМ[1], Л. БЕКБАЕВА[2], К. ОМУРБЕКОВА[1]
*[1]Институт Металлургии и обогащения, Алматы, Казахстан, *e.mail: elashmawi5@yahoo.com*
[2]Казахско-Британский технический университет, Алматы, Казахстан

СИНТЕЗ И ХАРАКТЕРИСТИКА АНТИКОРРОЗИОННЫХ ЭМУЛЬСИОНЫХ ЛАТЕКСОВ ДЛЯ МЕТАЛЛА

Резюме. Сополимерные эмульсионные латексы на основе стирола (St) и 2-этилгексилакрилата, (2-Эха) получены эмульсионной полимеризацией с различными соотношениями составов (80:20% и 50:50 %). Полимеризацию проводили при температуре 80 °C с использованием персульфата аммония (APS) в качестве инициирующей системы в присутствии сульфата додецилбензола натрия (DBS) в качестве поверхностно-активного вещества. Для решеток сополимеров характерны FT-IR, 1HNMR, TGA и DSC. Наличие нового пика поглощения в инфракрасной области C-H растяжения и C-H из плоского изгиба полистирола при 3027 и 696 $см^{-1}$ и химический сдвиг 1H ЯМР при 1.5-1.7 ppm из-за присутствия –CH_2– в сополимере показывает, что полимеризация имела место. Термогравиметрический анализ (ТГА) показал более высокую термическую стабильность, полученную при температуре разложения. Эти сополимеры имеют одиночную температуру стеклянного перехода, показывая, что эти сополимеры могут сформировать однородный участок. Однако с увеличением соотношения 2-этилгексилакрилата в сополимере повышалась температура стеклования. Результаты показали, что увеличение соотношения 2-Эха усиливает антикоррозионные свойства покрытых пленок. Полученные сополимеры показали отличные адгезионные свойства на металле. Результаты показали, что латекс сополимера имеет хорошую стабильность в коррозии, ультрафиолетового света, и может использоваться как антитикоррозионный металл. Также увеличение соотношения 2-Эха усиливает антикоррозионные свойства металла.

Ключевые слова: сополимер, эмульсия, 2-Эха, антикоррозионные свойства

Поступила 29.04.2018