



Acacia Gum as Protective Colloid and Stabilizer in the Polymerization of Vinyl Acetate



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Abstract

This study was directed to use the natural gum as a polysaccharide in the emulsion polymerization of vinyl acetate and vinyl ester of versatic acid monomers as an alternative material to hydroxyethyl cellulose, which is functioning as a stabilizer and protective colloid in the emulsion polymerization reaction of polyvinyl acetate copolymer. The solid materials content, pH, and viscosity of the prepared emulsion polymers with different contents of the polysaccharide were studied. The stability of the prepared emulsion polymers was studied. The scanning electron microscopy study of the prepared films from the prepared emulsion polymers using different concentrations of gum and hydroxyethyl cellulose, the minimum film-forming temperature, and the water absorption of the polymer films were investigated in detail. Gloss, wet scrub resistance, and hiding power of the paint samples that were prepared by 20% of the prepared polymers were studied to evaluate the difference between the natural gum and hydroxyethyl cellulose during the polymerization process and the effect of changing between gum and hydroxyethyl cellulose on the polymer properties and its application in the emulsion paint.

Keywords: polysaccharids; emulsion polymer; emulsion paints

1. Introduction

Polyvinyl acetate (PVAc) is one of the most common polymers. One possibility of producing PVAc at an industrial scale is protective colloid stabilizing emulsion polymerization. In such systems, the amphiphilic protective colloid forms a colloidal system that acts as a reaction center in which the polymerization process takes place [1-3].

Oligomers formed in the water phase from monomer molecules and the water-soluble initiators diffuse into the protective colloid micelles and react there to polymer chains with further monomer molecules diffusing into the micelle; emulsion polymerization provides numerous advantages, like being a greener process [4].

possessing low viscosity throughout the process, providing advanced heat transfer, and a narrower particle size distribution (PSD) than regular suspension polymerization processes [5].

In the industry, the control of the PSD is important to obtain specifically designed products, as the PSD determines the characteristics and properties of such dispersions [6].

Hydroxyethyl cellulose is used as a protective colloid in the polymerization of vinyl acetate copolymer. Hydroxyethyl cellulose is a polysaccharide with a linear chain of glucose molecules and the repeat unit consisting of two anhydroglucose rings connected by β -1,4-glucosides bond. Sources of cellulose are mainly softwood and hardwood, agricultural sources such as corn, jute, and sugarcane bagasse. From these sources, cellulose is extracted, isolated, and then modified for a specific application such as emulsion polymerization of ester derivatives and the paint industry Fig. 1 [7-10].

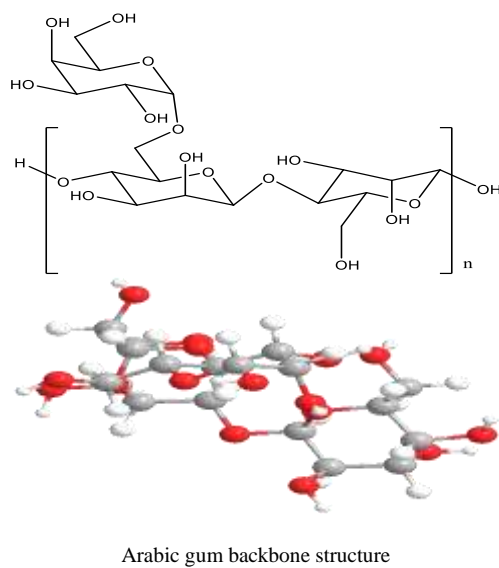
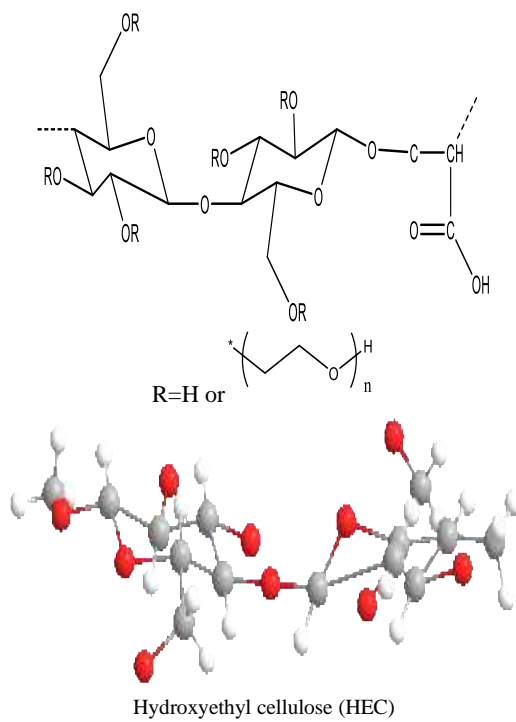
The global energy crisis and overconsumption of non-renewable resources have depleted natural resources, caused climatic changes with global warming, and raised the sea level. The research on alternative sources, chemicals, and the importance of the green materials usage increases every day.

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Receive Date: 18 February 2025, Revise Date: 20 March 2025, Accept Date: 31 March 2025

DOI: 10.21608/ejchem.2025.361104.11320

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Arabic gum in solid state

Fig. 1. The structure of HEC and Gum

Arabic gum is extracted from the stems and branches of certain species from the *Acacia* genus [11]. Due to the desirable properties, safety record, and natural origin, it is the most commercially valuable exudate gum, with wide applications in the industry as food, beverages, cosmetics, printing, ceramics, pharmaceuticals, photosensitive chemicals, textiles, paper, ink, paints, pyrotechnics, and adhesives [12, 13]. Recorded use of Arabic gum dates to 2000 B.C. by the ancient Egyptians in many fields like foodstuffs, hieroglyphic paints, and mummification ointments [14]. Arabic gum is a sustainable, biodegradable, food grade, and abundant source in nature, especially in Africa [15]. Surface hydroxyl groups in Arabic gum can act as sites for making it material for stabilizing polyvinyl acetate that is used as a binder for a wide range of plastic paints [16].

Arabic gum is a branched, neutral, or slightly acidic complex polysaccharide obtained as a mixed calcium, magnesium, and potassium salt. The backbone consists of 1,3-linked β -D-galactopyranosyl units. The side chains are composed of two to five 1,3-linked β -D galactopyranosyl units, joined to the main chain by 1,6-linkages. Both the main and the side chains contain units of α -L-arabinofuranosyl, α -L-rhamnopyranosyl, β -D-glucuronopyranosyl, and 4-O-methyl- β -D-glucuronopyranosyl, the latter two mostly as end-units Fig. 1 [17].

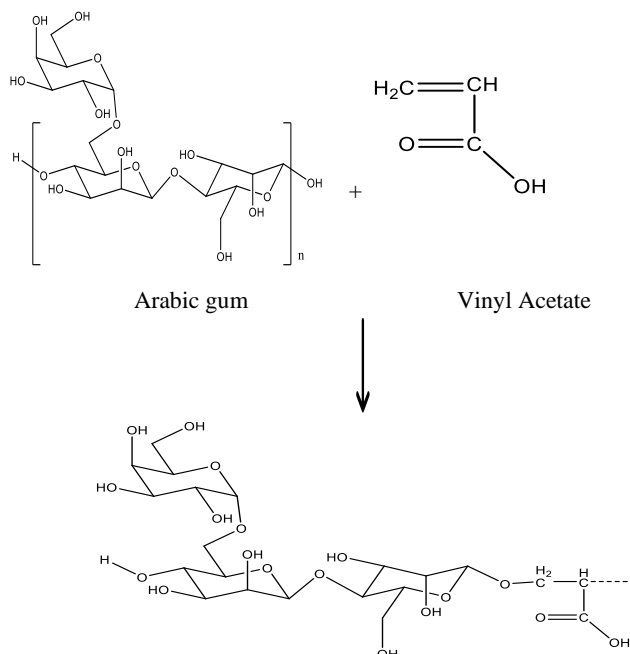
Arabic gum readily dissolves in cold and hot water in concentrations up to 50%. Because of the compact, branched structure and therefore small hydrodynamic volume, Arabic gum solutions are characterized by a low viscosity, allowing the use of high gum concentrations in various applications. Arabic gum has excellent emulsifying properties [18]. The hydrophobic polypeptide backbone strongly adsorbs at the oil–water interface, while the attached carbohydrate units stabilize the emulsion by steric and electrostatic repulsion. It was found that the stability of beverage emulsions is influenced by the number of processing factors, such as pasteurization and demineralization, and by the pH of the emulsion [19, 20].

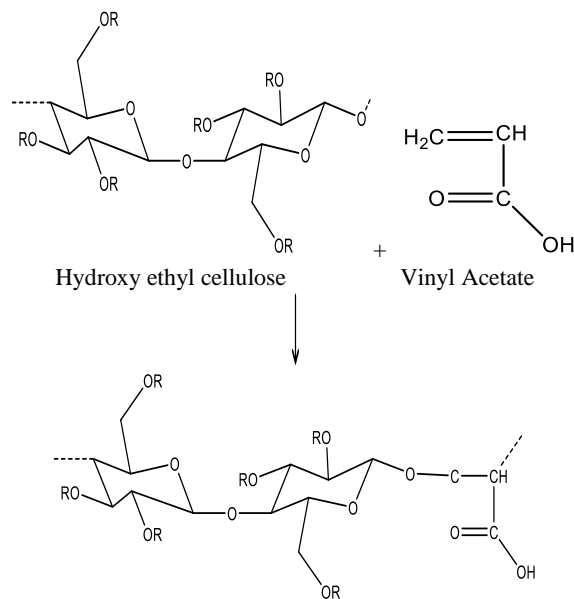
The current study aims to investigate Arabic gum as alternative material of hydroxyethyl cellulose as protective colloid and stabilizer in the polymerization of vinyl acetate and vinyl ester of versatic acid monomers using sodium persulphate as initiator and the application of the prepared polymers with different concentrations of Arabic gum and hydroxyethyl cellulose in the paint preparation and investigate the properties of the prepared paints.

2. Results and Discussion

2.1 Synthesis of the polymer

Emulsion polymerization systems hold significant importance in the chemical industry, particularly in the production of adhesives, sealants, and large-scale coatings. In the current study, we prepared five new polymers depending on changing the percentage of both AG and HEC while fixing the quantities of other added materials such as water, Veova, VAM, and sodium persulphate (Table 1). The reaction was proceeded via an addition reaction between AG and/or HEC with the vinyl acetate monomer and vinyl ester of the Versatic acid monomer, Schemes 1 and 2.



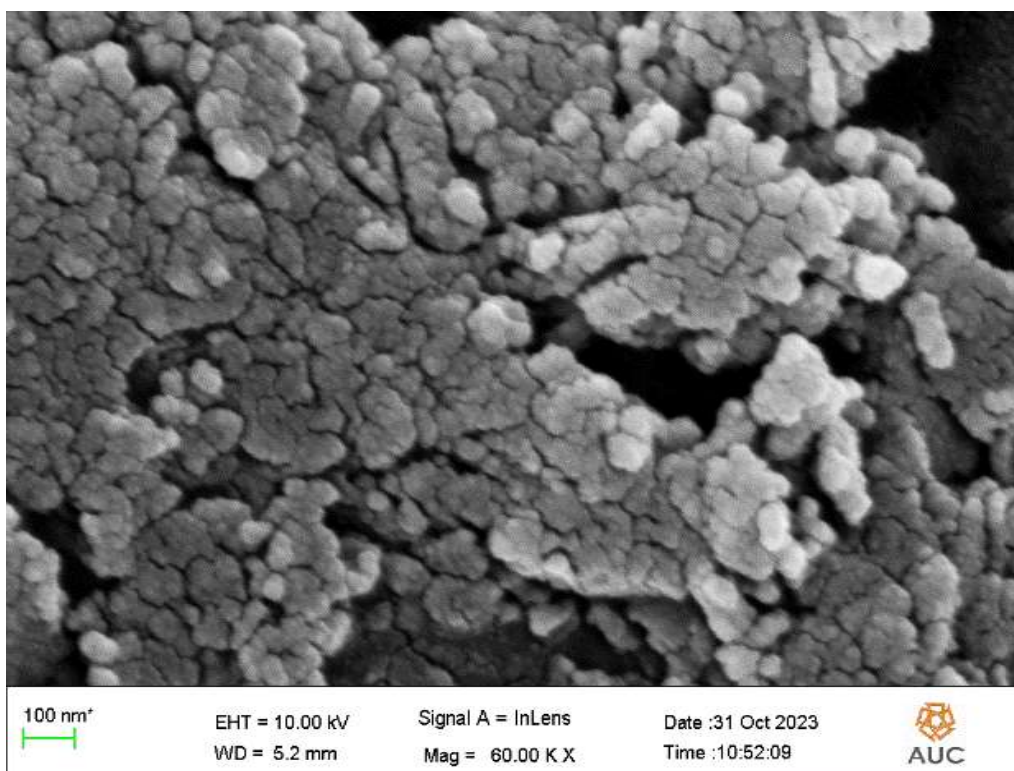


Scheme 2

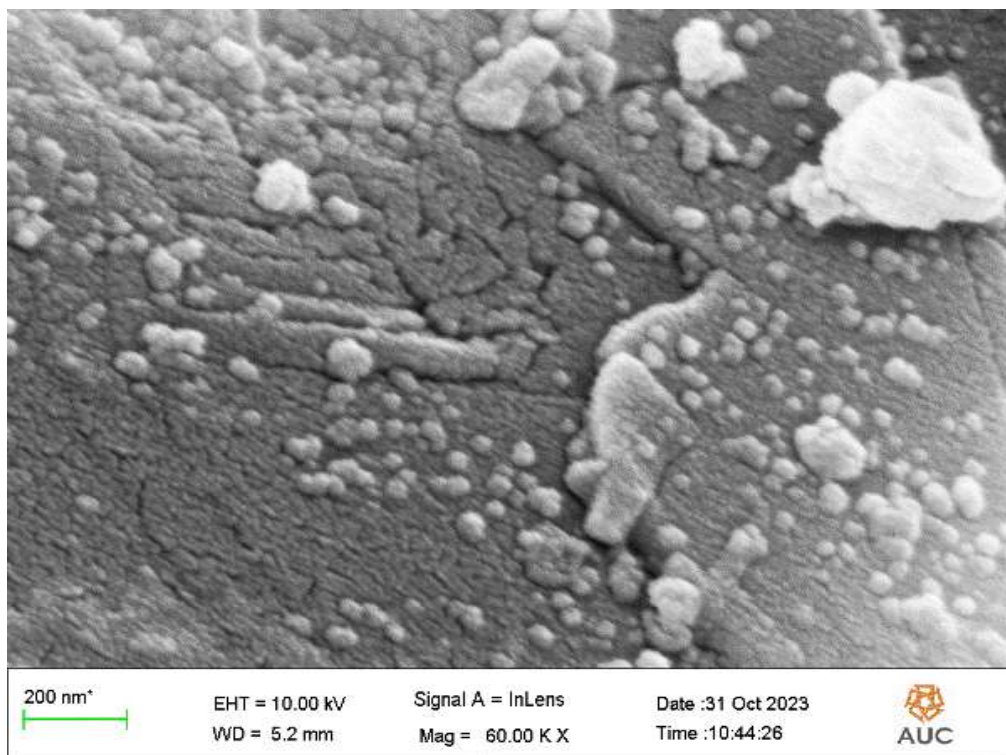
2.2 Characteristics of the Polymer

2.2.1 Morphology of the polymer

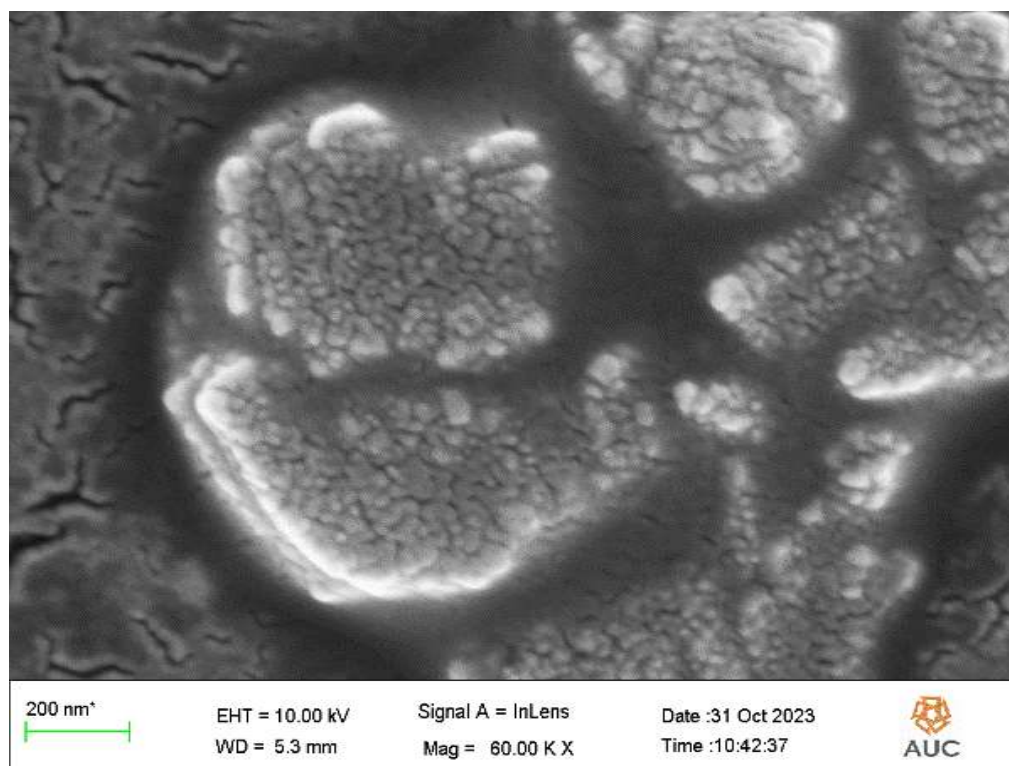
Formed films for all the polymer samples are transparent, and the morphological structure of the prepared polymers was studied via SEM (Scanning Electron Microscope) is shown in Fig. 2. The results indicated that the film of the polymer prepared by 40% AG and 0% HEC has the appropriate particle size and the uniform particle distribution.



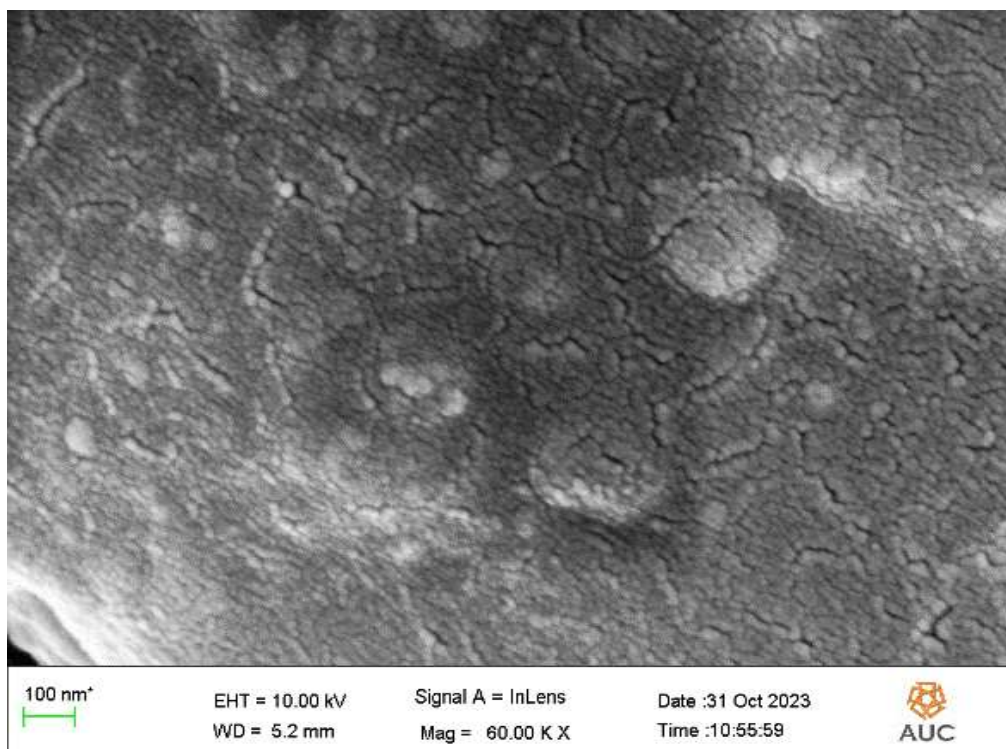
SEM micrograph of film 500 μ from sample 1 (40% HEC + 0% AG)



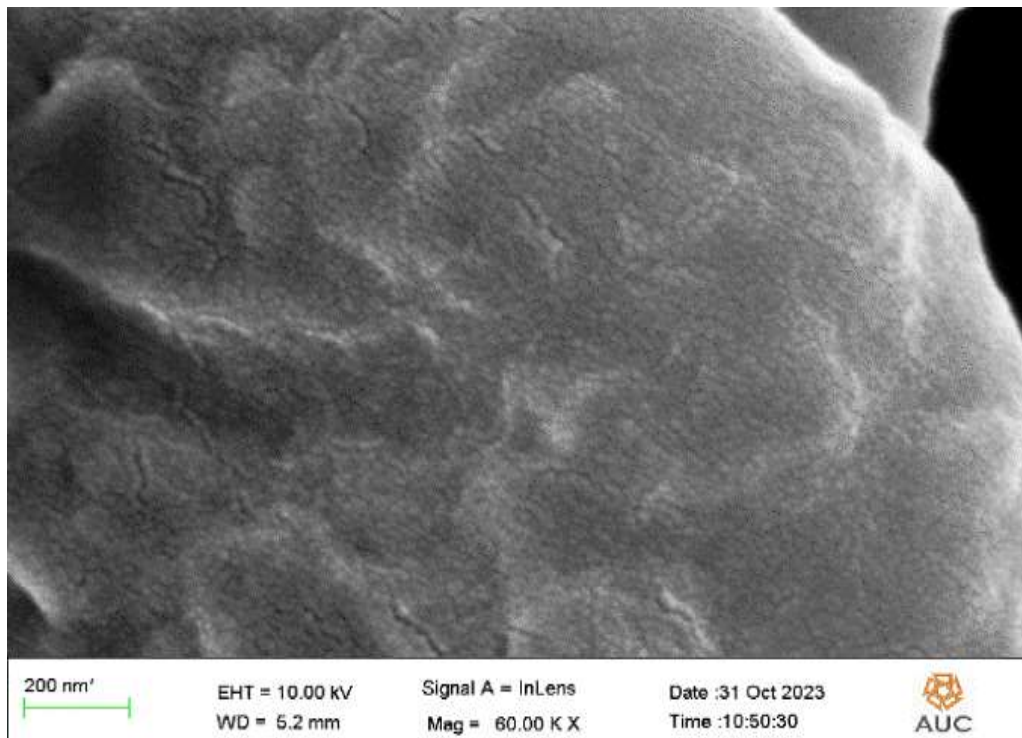
SEM micrograph of film 500 μ from sample 2 (30% HEC + 10% AG)



SEM micrograph of film 500 μ from sample 3 (20% HEC + 20% AG)



SEM micrograph of film 500 μ from sample 4 (10% HEC + 30% AG)



SEM micrograph of film 500 μ from sample 5 (0% HEC + 40% AG)

Fig. 2. SEM micrograph of polymer samples prepared by different content of polysaccharides

2.2.2 pH of the emulsion polymer samples

Device name: Metrohm 827

Method Reference: ASTM D4317-9.1.4

pH was measured for the polymer samples after 24 hours and 3 months from the polymerization, as shown in Fig. 3.

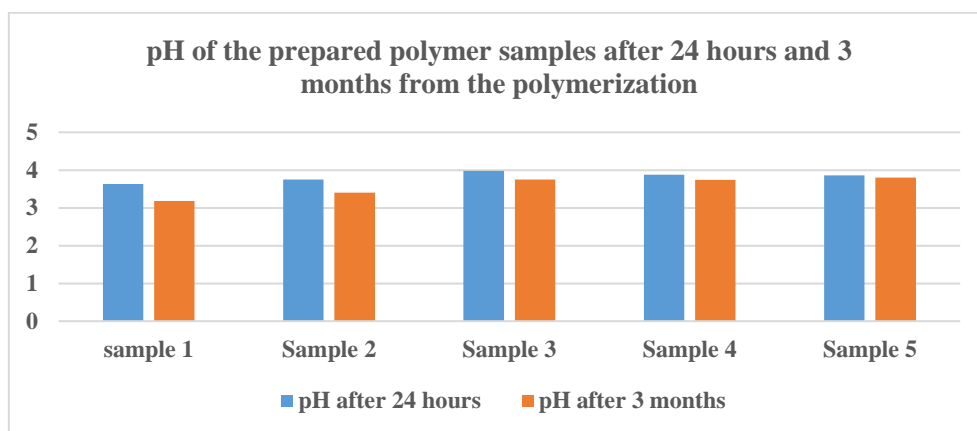


Fig. 3 pH of the prepared polymer samples after 24 hours and 3 months from the polymerization

2.2.3 Viscosity of the emulsion polymer samples

Device name: Dial Reading BROOKFIELD Viscometer

Method Reference: ASTM D4317-9.1.1

Viscosity was measured for the polymer samples after 24 hours and 3 months from the polymerization, as shown in Fig. 4.

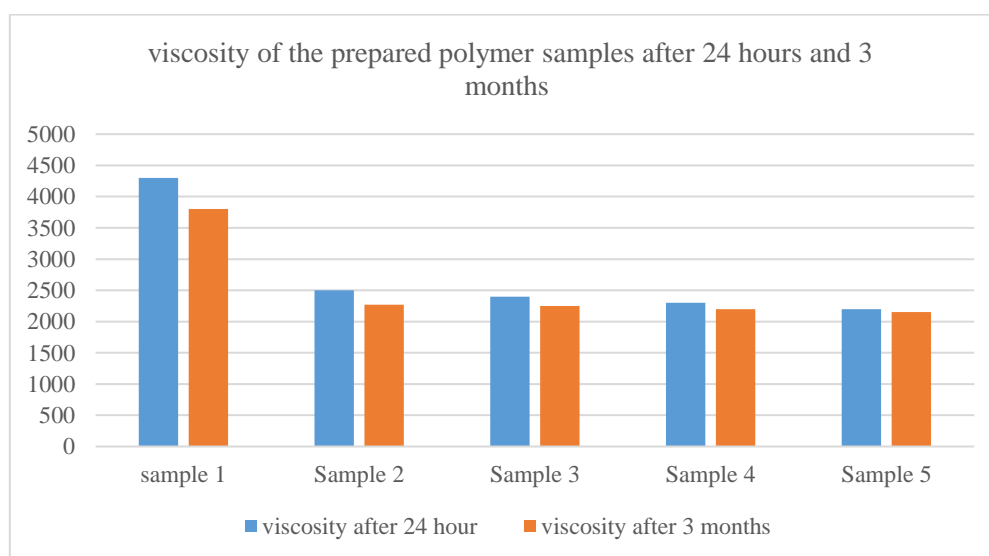


Fig. 4 viscosity of the prepared polymer samples after 24 hours and 3 months from the polymerization

2.2.4 Solid content of the emulsion polymer samples

Method reference: ASTM D2834

Solid materials content in the emulsion polymer was measured for the polymer samples after 24 hours and 3 months from the polymerization as shown in Fig. 5.

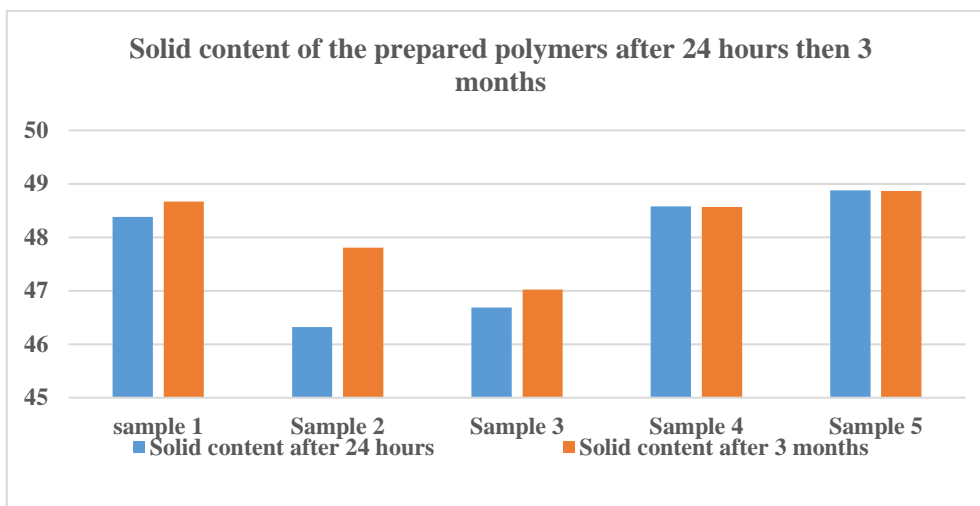


Fig. 5 Solid content of the prepared polymers after 24 hours and 3 months from the polymerization

2.2.5 Emulsion polymers stability

Viscosity of the emulsion polymer was measured after 24 hours from the polymerization and measured again after three months to compare and evaluate the stability of the emulsion polymers with different polysaccharide contents. The stability was improved by increasing the Arabic gum in the polymer as shown in Fig. 6 due to the presence of the pyranosyl ring, as shown in Fig. 1. The reason is improving the particle size distribution of the polymer by increasing AG in the polymer and the different types of branching in HEC and AG.

$$\text{visc. stability} = \frac{\text{viscosity after 3 months}}{\text{viscosity after 24 hours}} \times 100$$

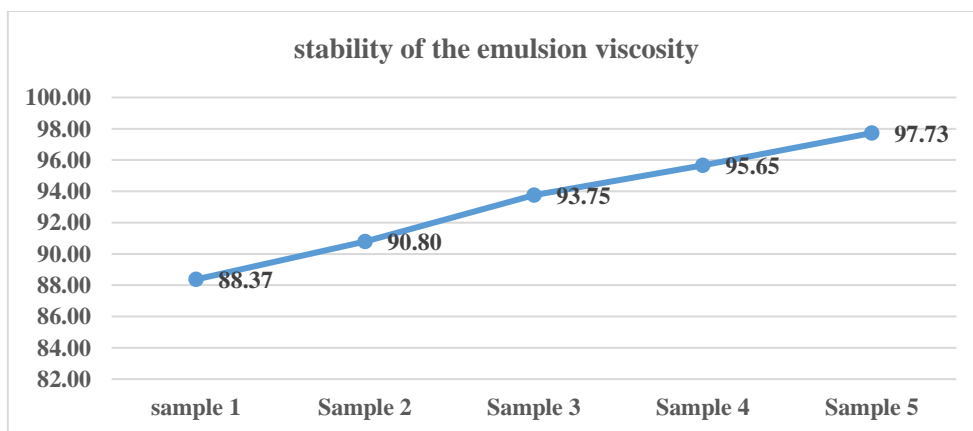


Fig. 6 stability of the emulsion viscosity

2.2.6 Minimum film forming temperature (MFFT)

Device Name: RHOPOINT instrument
Method Reference: ASTM D2354

Minimum film-forming temperature was measured for the polymer samples, and results showed minor changes by replacing HEC with AG, as shown in Fig. 7.

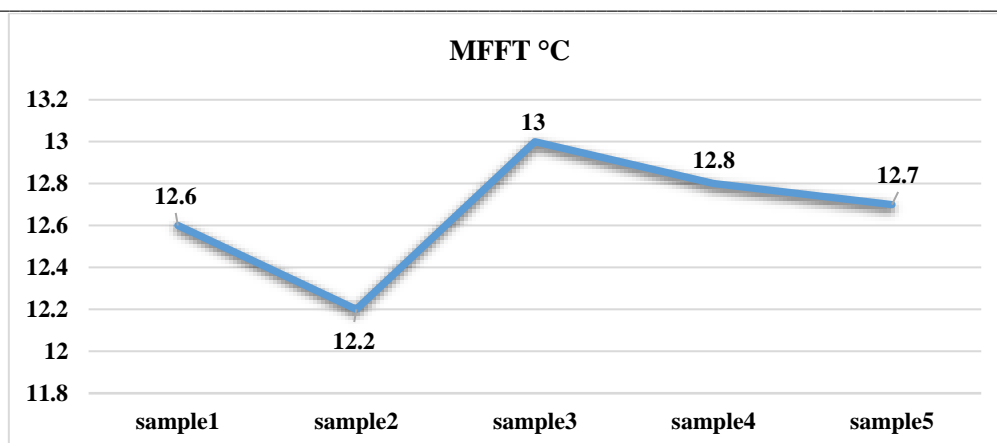


Fig. 7 MFFT of the prepared polymer samples

2.2.7 Polymer Water Absorption

The water absorption by the polymer film of 500 μ was measured for the prepared samples by immersing the film in distilled water for 24 hours at 24°C.

$$\text{water ab.} = \frac{(\text{wet weight} - \text{dried film weight})}{\text{dried film weight}} \times 100$$

Water absorption of the polymers that were prepared by 100% of HEC solution and 100% of AG solution are similar but less than in the polymers that were prepared by mixed solutions of HEC and AG, as shown in Fig. 8.

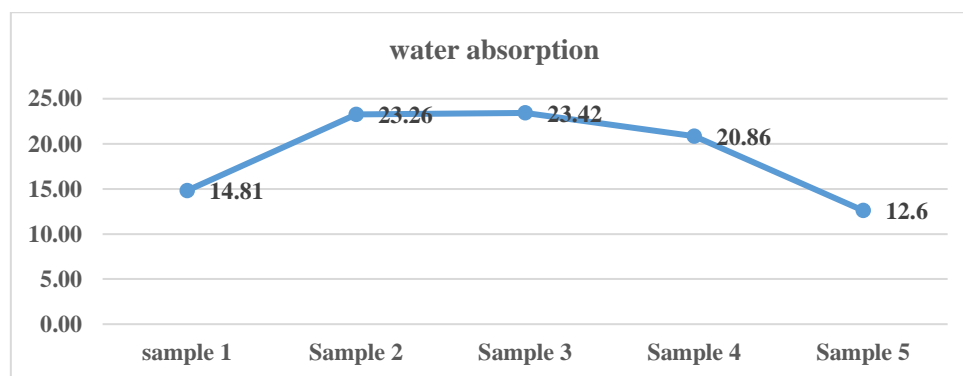


Fig. 8 water absorption of the prepared polymer samples

2.3 Preparation of emulsion paints

The paint samples were prepared by adding 20% of the prepared emulsion polymers to the emulsion paint base to prepare five samples of paints.

2.3.1 Wet scrub resistance of the paint films

Device name: Elcometer 1720
Method Reference: ASTM D2486

Wet scrub resistance was measured for the samples of the paints that were prepared by adding 20% from prepared emulsion polymers. Wet scrub resistance was improved by increasing AG percent in the polymer as shown in Fig. 9. The reason is improving the particle size distribution by increasing AG in the polymer and the different types of branching in HEC and AG.

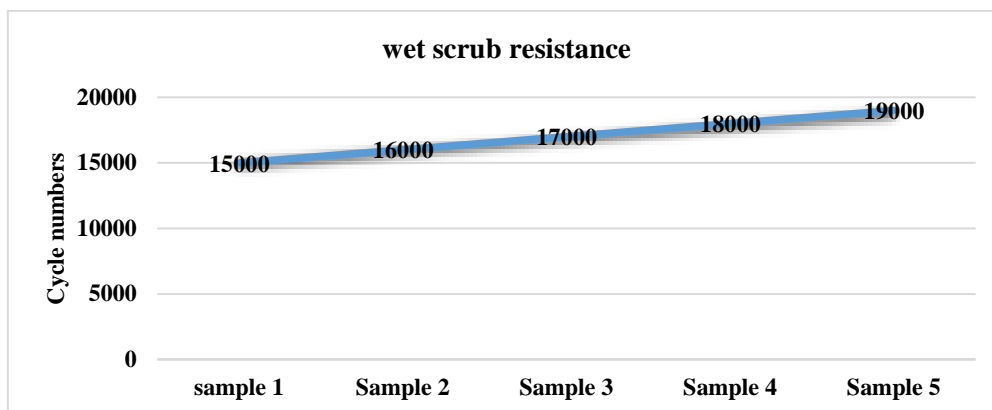


Fig. 9 Wet scrub resistance of the prepared paint samples

2.3.2 Gloss of the paint films

Device name: Elcometer 401 Novo-Gloss

Method Reference: ASTM D2457

Gloss was measured for the samples of the paints prepared by 20% of the emulsion polymers. Gloss was not changed by replacing HEC with AG and showed lower values by the polymers containing a mixed solution of HEC and AG, as shown in Fig. 10.

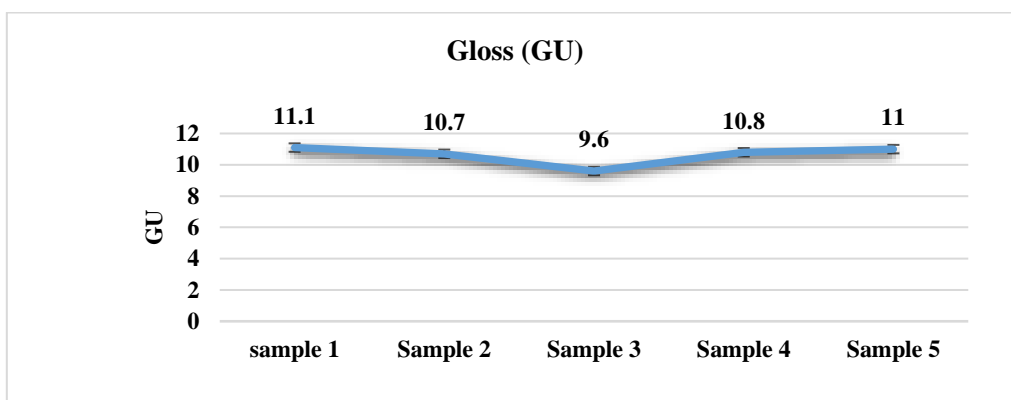


Fig. 10. Gloss of the prepared paint samples

2.3.3 Hiding power and opacity of the paint films

Device Name: Sheen Opac 310 Reflectometer

Method Reference: ASTM D2805

Hiding power and opacity were measured for the samples of paints prepared with 20% of emulsion polymers with different content of polysaccharides. Contrast ratio is the most common way of measuring opacity in the paint industry.

Table 1. Contrast ratios of the prepared emulsion paints

	84.2/91	86.1/90.6	85.3/90.1	83.5/89.7	85.1/91.2
Difference	6.8	4.5	4.8	6.2	6.1
Contrast ratio	92.5	95	94.7	93.1	93.3

Hiding power and opacity showed minor changes in the paints prepared by the polymers of different content of polysaccharides as shown in **table 1**.

3. Experimental

3.1 Materials

The materials that were used in this study were supplied by VAM (Dow), Veova (Hexion), HEC (Ashland), sodium persulphate (Sisco), and Gum (Kapadia Gumchem Industries). The produced emulsion polymers were studied via scanning electron microscope (American University in Cairo). Polyvinyl acetate copolymer with vinyl ester of Versatic acid was prepared using 40% of polysaccharide solution (3% in soft water) as protective colloid, viscosity stabilizer, and emulsifier. All experimental characterizations were conducted according to ASTM D4317-9.1.1, D4317-9.1.4, ASTM D2834, ASTM D2354, ASTM D2486, ASTM D2457, and ASTM D2805.

3.1.1 Gum solution 3% in water

Arabic gum was dissolved in hot soft water at 95°C to prepare 3% solution. The AG solution was filtered on filter paper to be used in the preparation of polyvinyl acetate copolymer by different percentages in the reaction with HEC solution 3% to understand the effect on the thermal profile of the reaction, the final product properties, and its application in the preparation of emulsion paint [21-24].

3.1.2 Hydroxyethyl cellulose (HEC) 3% in water

The solution 3% of hydroxyethyl cellulose was prepared in soft water at room temperature to be used in different percents with Arabic gum solution 3% in the emulsion polymerization of vinyl acetate with vinyl versatate copolymer.

3.1.3 Sodium persulphate

A solution of sodium persulphate was prepared 10% in soft water at room temperature.

3.2 Methods

3.2.1 Preparation of the polymer

Five samples were prepared by emulsion polymerization of vinyl acetate monomer with vinyl ester of versatic acid using different percent of hydroxy ethyl cellulose 3% solution and Arabic gum 3% solution. Sodium persulfate 10% was added as an initiator [25, 26].

Table 2. composition of the prepared polymer samples

Raw Material	Sample1	Samle2	Sample3	Sample4	Sample5
HEC 3% in water	40%	30%	20%	10%	0%
AG 3% in water	0%	10%	20%	30%	40%
H ₂ O	5%	5%	5%	5%	5%
Veova 10	8%	8%	8%	8%	8%
VAM	41%	41%	41%	41%	41%
Sodium Persulfate 10% in H ₂ O	6%	6%	6%	6%	6%

3.2.2 Chemical methodology

In the glass reactor of volume 1.8 L, the emulsion polymer was prepared in five different reactions with the different components of polysaccharides solutions as shown in **Table 2**.

3.2.3 Polymerization reaction

Polysaccharide solution was added in the reactor, and mechanical stirring of the solution at 300 rpm was followed by the addition of water. The addition of the monomers (VAM, Veova10) and the initiator (sodium persulphate 10%) in the reactor for 5 hours with measuring the temperature of the reaction mass. with continuous stirring of the reactants at 300 rpm. When the temperature of the reactor reached 78°C, the cooling of the reactor started to keep the temperature below 85°C. Addition of the initiator was continued for 10 minutes after complete addition of monomers. The reaction mixture was cooled to below 50°C. the product was discharged from the reactor.

4. Conclusion

The SEM study of the prepared polymers showed that the polymer, which was prepared by natural AG, is more homogenous with similar particle size and distribution. The stability of the prepared emulsion polymers was measured by measuring the deviation in the viscosity after three months. The results showed that the stability was increased by increasing the AG% in the prepared emulsion polymer. The minimum film-forming temperature of the polymer films was measured, and the results showed minor changes for the samples by HEC and natural AG. Water absorption of the dried films of the polymers was lower in the polymer prepared by the natural AG. The emulsion paint samples were prepared by 20% of the polymers, and the wet scrub resistance test showed slight improvement in the samples with AG. The gloss of films for paint samples was almost similar. The contrast ratio was measured for the paint samples; the results are similar with a slightly increase in the samples which contain mix of AG and HEC solutions in the polymer.

5. Conflicts of interest

There are no conflicts to declare.

6. Formatting of funding sources

Corresponding author is responsible for funder's requirements.

7. Acknowledgment

I am highly thankful to chemistry department of Suez university for the active guidance throughout the completion of the project. Last but not the least, I would want to extend my appreciation to those who could not mentioned here but here played their role to inspire the curtain.

Abbreviations

HEC	Hydroxy ethyl cellulose
AG	Arabic gum
PSD	Particle size distribution
MFFT	Minimum film forming temperature
VAM	Vinyl acetate monomer
Veova	Vinyl ester of Versatic acid
PVAc	Polyvinyl acetate

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