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Polysaccharides as Protective Colloid and Stabilizer in the Polymerization of Vinyl Acetate

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ABSTRACT

Numerous experiments have been conducted on the emulsion additional polymerization of vinyl acetate (VAc) copolymers, such as replacing polyvinyl alcohol with renewable materials such as hydroxyethyl cellulose (HEC), starch or natural gum as protective colloids or adding renewable materials to the emulsion as additives or fillers during or before the polymerization. These experiments synthesized polyvinyl acetate (PVAc) copolymers from vinyl acetate monomer (VAM) as raw material and potassium or ammonium persulfate as inorganic thermal initiators in aqueous media containing various protective colloids to improve the properties of the formed polyvinyl acetate (PVAc) emulsions. Physically dispersed titanium dioxide as cross-linking agent in the polymer hydrogels were prepared by a one-step emulsion free radical polymerization mechanism using acrylic acid (AA) as comonomer, Arabic gum (AG) as protective colloid and iron ions (Fe^{3+}) as crosslinking agent. In these experiments, the polymerization process was tested by varying the amount of protective colloid, crosslinking agent or dispersant. The polymers were tested for properties such as morphology, stability, tensile strength and hardness, and some applications of the obtained polymers were tested to check their performance..

1. Introduction

Polyvinyl acetate (PVAc) is an important polymer used in the adhesive and coating industries. In the industrial production of PVAc, protective colloid emulsion polymerization can be used. The protective colloid acts as a reaction center in which the polymerization reaction takes place [1-3]. The monomer molecules begin to form micelles in the aqueous phase, and the water-soluble initiator interacts with the micells of the protective colloid and binds to the growed chains in the polymer, while the monomer molecules diffuse into the micelles. Aqueous emulsion polymerization has many benefits, such as green process [4], low viscosity, excellent heat transfer, and similar distribution in the particle size (PSD) range compared to suspension polymerization processes [5]. In industry, the PSD range is crucial to obtain a specific product because the PSD detects the properties of the polymer [6]. Polyvinyl alcohol (PVA) is used as a protective colloid and stabilizer in the emulsion polymerization of vinyl acetate (VAc). The highest effective polymeric stabilizers are amphiphilic block copolymers or graft copolymers. The insoluble part of the polymer settles the stabilizing soluble groups on the colloidal particles.

When two or more particles collide, the repelling groups escape from the repelling zone either by adsorption or by external movement on the chain surface. Effective settling prevents the repelling and imparts stabilization through permeation and bulk repulsion. average molecular weight (77,100-79,100 gmol1), hydrolyzed PVA (<90%), and block PVA are effective in stabilizing polyvinyl acetate (PVAc) latexes [7-9]. The hydrophobic PVAc chains support the main settling moiety, and the hydrophilic part of PVA chains enters the aqueous phase as stabilizing blocks.

Hydroxyethyl cellulose (HEC) is used as a protective colloid and stabilizer in the polymerization of vinyl acetate copolymers. Hydroxyethyl cellulose is a linear polysaccharide composed of glucose molecules, the repeating unit of which contains two rings of anhydroglucose linked by β-1,4 glycosidic bonds. Cellulose comes from softwoods and hardwoods, as well as agricultural resources such as corn, bagasse, and fruit trees. Cellulose is extracted from these sources, separated, and then modified for use in applications such as emulsion polymerization of ester derivatives and the coatings industry [10-12].

The over-consumption of non-renewable resources has exacerbated the global energy crisis and brought about major challenges, the most critical of which include climate change caused by global warming and rising sea levels.

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The research on alternative sources of chemicals and the use of green materials are becoming increasingly important.

Arabic gum is extracted from the trees stems and branches of certain species of the Acacia genus [13]. Due to its reliable properties, safety and natural origin, arabic gum has become a valuable natural material and is widely used in industries such as food, cosmetics, printing, ceramics, medicine, chemicals, textiles, papermaking, coatings and adhesives [14,15]. As early as 2000 BC, the ancient Egyptians began to use arabic gum and applied it to various fields such as hieroglyphic paints, food and mummy ointments [16]. Arabic gum is a sustainable food-grade, biodegradable natural resource with abundant reserves in nature, especially in Africa[17]. The surface hydroxyl groups of arabic gum can be used as a raw material to stabilize polyvinyl acetate, which in turn can be used as an adhesive for various plastic coatings[18]. Arabic gum is a branched neutral polysaccharide or slightly acidic, extracted in the form of a mixed salt of magnesium, calcium and potassium. The main chain structure is composed of 1,3-linked β-D-galactopyranosyl units. The side chains consist of 2 to 5 1,3-linked β-D-galactopyranosyl units, which attache to the backbone via 1,6-linkages. The backbone and branched chains contain α-L-arabinofuranosyl, α-L-rhamnosyl, β-D-glucopyranosyl, and 4-O-methyl-β-D-glucopyranosyl units, the las two are the end units [19]. Arabic gum is good soluble in hot and cold water at concentrations up to 50%. Due to its branched structure, the hydrodynamic volume is small and the viscosity of arabic gum solutions is low, making it easy to use at high concentrations in a variety of applications. Arabic gum is good emulsifier [20]. The hydrophobic peptide structure adsorbs at the water-oil interface, while the linked carbohydrate units help in stabilizing the emulsion through steric hindrance and electrostatic repulsion, resulting in emulsion stability that is enhanced by a variety of processing factors (e.g., demineralization) and the pH of the emulsion [21,22].

2. Literature review

2.1 Polyvinyl alcohol stabilized polyvinyl acetate emulsion

Water-soluble polymers, especially polyvinyl alcohol (PVA), have attracted much attention in the coatings and adhesives industry due to the growing demand for solvent-free adhesives. These adhesives are favored because they do not emit volatile organic compounds (VOCs) and are more environmentally friendly.

PVA is essential to produce organic solvent-free adhesives. Its controlled degree of chain length and hydrolysis (DH) improve its performance. PVA has excellent rheological properties and is suitable for porous materials, especially for wood adhesives.

PVA is used as a protective colloid and stabilizer in the emulsion polymerization of vinyl acetate to produce polyvinyl acetate (PVAc). This application improves the mechanical properties of the resulting adhesive. The molecular weight, degree of hydrolysis (DH) and particle size distribution of PVA are key factors in determining the colloid stability in adhesive formulations.

Polyvinyl acetate emulsions prepared with polyvinyl alcohol as a colloidal protective agent and emulsifier material have been widely used in wood and paint adhesives, when vinyl acetate is copolymerized with other monomers such as dibutyl maleate, ethylene, ethyl acrylate, methyl acrylate or 2-ethylhexyl acrylate, vinyl laurate and versatate (Veova). A large number of experiments have been conducted on the emulsion polymerization of VAc using PVA as an emulsifier. In the previous studies, the reaction process of the vinyl acetate free radical polymerization differs from the standard scheme of the Smith Ewart theory due to the water solubility of the monomers (2.5 wt% at 25°C and 3.5 wt% at 70°C) and the chain transfer constants to the monomers and the formed polymer. Therefore, the vinyl acetate polymerization starts in the water phase and the free radicals will continue to grow until they reach a size that exceeds their water solubility range and precipitate in the solution to form primary polymer particles and accumulate with existing particles. The process feature that enhances the vinyl acetate polymerization reaction kinetics to differ from its standard model to be grafted and that occurs between PVA and VAc during the polymerization reaction.

Hartley documented the formation of the grafting processes by fractionation techniques, where two parts were noted: one that was water-soluble and high in PVA, and another that was water-insoluble and contained grafted PVA. The grafting process resulted in chain transfer leading to a decrease in free radical reactivity, resulting in a decrease in the polymerization rate. Different degrees of grafted PVA were observed by extracting the films of the polymers in water and benzene. Using PVA as a stabilizer, the acetate groups of PVA grow during the polymerization, resulting in polymer chains with higher emulsifying properties. In addition, the amount of PVAc that is grafted on PVA in the radiation-initiated polymerization was evaluated and assessed by estimating the weight gain of the PVA polymer by acetone extraction.

2.2 Polymerization Process using polyvinyl alcohol as protective colloid:

The emulsion polymerization was conducted in a one liter three nozzols reactor. The agitation was started at 100-130 rpm using a half moon stirring blade fixed on a shaft inserted into the glass container. A long-shaped condenser was connected to minimize monomer vapour loss. A sampling valve was used to periodically collect latex samples, which consisted of a nozzole connected to a valve for continuous injection of nitrogen to blanket the space above the reactants. The reactor was cooled using a water bath at a constant temperature of 60-65°C; 700 g of polymer was prepared for each experiment.

A typical recipe for seed latex preparation in VAM emulsion polymerization was used as a standard for all experiments. The recipe is shown in Table 1, based on 100 g of polymer.

Table 1. standard recipe used in batch emulsion polymerizations of vinyl acetate using polyvinyl alcohol as emulsifier

Material	Weight (g)
Distilled-deionized water	89.259
Polyviny1 alcohol (PVA)	3.445
Vinyl acetate (VAM)	7.275
Sodium bicarbonate ($NaHCO_3$)	0.007
Potassium persulfate ($K_2S_2O_8$)	0.014

100 g of latex was prepared by dissolving 3.445 g of PVA powder in demineralized water by a magnetic stirrer at room temperature. The solution was then heated to reach temperature of 95°C and the temperature was kept for 30 min constant to allow the PVA to completely dissolve. Then filtering the solution using a 45 μm stainless steal mesh, chilled water was used to cool the solution to room temperature, and then the solid materials was measured. During the polymerization reaction, addition of PVA solution to the polymerization flask was satrted, and the solid material was modified to compensate for the lose of water during PVA solution preparation. Purging of the reaction flask was with nitrogen for 30 min was important and then heating to 60°C. When the polymerization temperature was reached, 7.275 g of VAc and 1 g of 1.85 wt % sodium bicarbonate solution were added to the flask through a three way valve connected to a tube which was used as a sampling valve. 1 g of 1.85 wt % of potassium persulfate solution after 15 minutes was added; this was the zero point of the polymerization reaction. During the reaction, samples were collected periodically using a syringe, collected in bottles consisting of 1-2 drops of 1% solution of hydroquinone to terminate the reaction, the bottles were sealed with stoppers made of rubber, and stored at 4°C. The polymerization kinetics were followed, the conversion was assessed gravimetrically by collecting at least 30 g of samples.

In the polymerization of vinyl acetate emulsion using PVA as a colloid, micelles of different molecular weights are formed, which form in the water phase. Studies on repeated centrifugation of the redispersed precipitate indicated that not all water soluble species can be separated in a single centrifugation step. By using a new separation technique that allows the separation of polymers directly from the latex, the grafting of VAc onto PVA polymer molecules in the polymerization of VAc with PVA as a protective colloid and stabilizer was successfully studied and evaluated. The results show that acetonitrile selective dissolution of the polymer using a rotary evaporator is a feasible technique for the quantitative separation of PVAc homopolymers from water-soluble and water-insoluble PVA. Hot water selectively dissolves the insoluble part of acetonitrile, and the water soluble part of PVA can be separated from the grafted water-insoluble PVA part, which does not contain any impurities (i.e., PVAc micelles). In addition, this technique can also be used to determine the PVAc content in the grafted PVA [23].

2.3 Starch stabilized polyvinyl acetate emulsion

Non-renewable resource issues:

The growing global energy crisis has prompted research into alternative resources, particularly biomaterials.

Properties of starch:

Starch is an important biomaterial, making it an attractive option.

Cost-effectiveness: It is easily soluble in water and inexpensive. It can be used in adhesives, paper coatings, and textile applications [24].

Composition:

- Starch consists of two polysaccharide components:
- Amylose: a linear structure.
- Amylopectin: a branched structure.

The ratio of amylopectin to amylose varies depending on the starch source and contains crystalline and amorphous regions. The crystalline regions are stabilized by hydrogen bonds, which affects reactivity. It forms a gelatinous paste when heated in water and can be subjected to various chemical modifications (esterification, oxidation, etherification, cross-linking) [25]. Relevant techniques include ultrasonic degradation, wet heat treatment, and microwave treatment. Starch can be blended with polyvinyl acetate (PVAc) to partially or completely replace petrochemical-based polymers including polyvinyl alcohol (PVA) [26].

Grafting Polymerization:

Grafting vinyl acetate onto starch can enhance its thermal stability, mechanical strength, and water resistance. This approach has attracted the attention of researchers and the industry for its application in wood adhesives [27,28].

The natural abundance, versatility, and modification potential of starch make it a promising biomaterial for the development of sustainable adhesives, which can effectively replace non-renewable petrochemical resources. Further research and innovations in processing technology can enhance its application in various industries [29-34].

2.4 Cellulose-Based Wood Adhesive

Overview of Cellulose:

Definition: Cellulose is a biopolymer composed of linear chains of glucose molecules. It is a polysaccharide containing two anhydroglucose rings linked by β 1-4 glycosidic bonds to form a repeating unit [35,36].

Sources:

- Softwoods
- Hardwoods
- Agricultural sources (e.g., corn, jute, bagasse)

Extraction Process: Cellulose is extracted and separated from its source for specific applications [37-40].

Cellulose morphology:

Microcellulose: includes microfibrillated cellulose (MFC) and microcrystalline cellulose (MCC) [41].

Nanocellulose: includes cellulose nanofibers (CNF), and cellulose nanocrystals (CNC), and its size is in the nanometer range, as shown in **Fig. 1** [42-45].

Functionalization and performance:

The hydroxyl groups present in nanocellulose enhance its functionalization potential, thereby improving its adhesive properties [46-48].

Interaction: Since the size of nanocellulose monomers is smaller than that of polymer chains, the interaction repulsion between nanocellulose and monomers is smaller, thereby improving the dispersion of composite materials and reducing agglomeration [49-52].

Polymerization process:

Aqueous medium: The polymerization reaction in an aqueous medium containing dispersed nanocellulose is more efficient and is conducive to the interaction of polymer particles during the synthesis process. Nanocellulose can change the properties of adhesives [53,54].

Performance improvement: Improves the mechanical, physical and thermal properties of wood boards [55].

Environmental benefits:

Replacing synthetic adhesives with adhesives containing nanocellulose can reduce formaldehyde emissions [56-57].

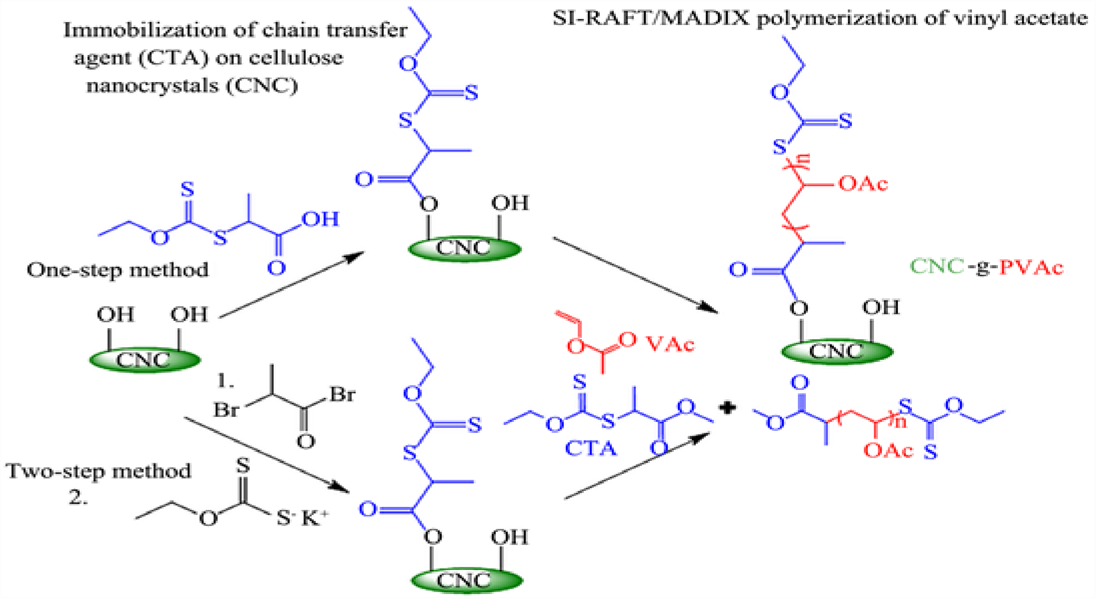

Fig. 1 CNC Grafted PVAc

2.5 Using Arabic Gum in Free Radical Polymerization.

Physically cross-linked composite hydrogels were prepared by free radical addition polymerization. Titanium dioxide (TiO_2) was physically dispersed in the hydrogel, which was synthesized by a single-step method with acrylic acid (AA) and Arabic gum (AG) as the main polymer components and iron ions (Fe^{3+}) as cross linkers.

Upon addition of ammonium persulfate (APS), the vinyl groups of AA polymerized and initiated the formation of a physically cross-linked hydrogel network. The low pKa values of AA (4.2) and AG (4.4) activated the carboxyl groups of both components, while the hydroxyl groups of AG were partially charged (–O−σ–H+σ). This interaction led to the formation of mono, di, and tri-ionic species containing Fe^{3+}.

In addition, the hydroxyl groups on AG promoted hydrogen bonding between itself and the carbonyl groups (-C=O) on the polymer chain. TiO_2 also contributed to the composites through physical interactions with AA and AG as shown in **Fig. 2**. Furthermore, the long polymer chains of AG enable it to physically entangle with AA, thus enhancing the impartiality of the network, and evaporation indicating good stability. After drying, the water content of the hydrogel was 2-5% without further

2.5.1 Hydrogel Preparation Procedure

Materials used:

- Arabic gum (AG)
- Ammonium Persulfate (APS)
- Titanium Dioxide (TiO_2) Powder
- Ferric Chloride ($FeCl_3$)
- Acrylic Acid (AA)
- Milli-Q Distilled Water

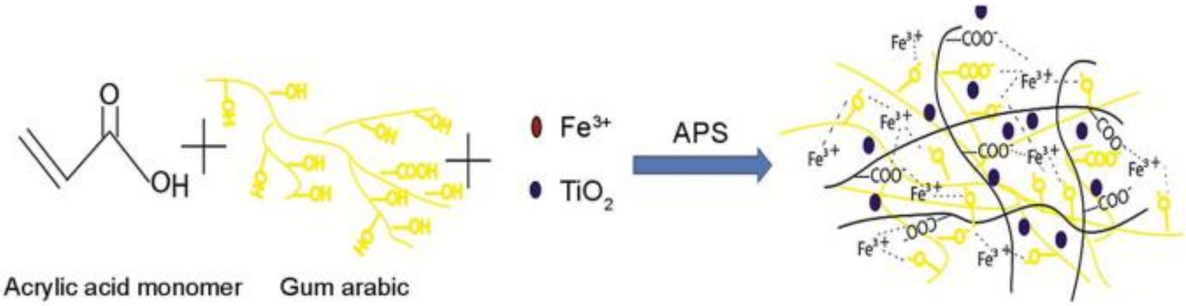

Fig. 2: The formation of polymer using Arabic gum

Preparation of Stock Solution:

- Dissolve 4 g of AG in 80 mL of demineralized water to form a homogeneous solution.

Hydrogel Synthesis:

- Take 10 mL of solution in a reaction vessel.
- Add 2 mL of AA.
- Mix in different amounts of TiO_2 powder (0.01 g, 0.02 g, and 0.03 g).
- Add 2.5% $FeCl_3$ (based on the total weight of AG, AA, and TiO_2) and stir for 2 hours.

Polymerization Initiation:

- Add 0.05 g of APS to initiate free radical polymerization.
- Pour the mixture into a pipette and bake in an oven at 40°C for 2 hours.

Final steps:

- Remove the sample from the pipette, place in a petri dish and dry at room temperature.

Synthesis and numbering of hydrogels:

Synthesized hydrogels:

- AA–AG is numbered AG containing TiO_2 (AGT1, AGT2, AGT3), where:
- AGT1: 0.01 g TiO_2
- AGT2: 0.02 g TiO_2
- AGT3: 0.03 g TiO_2

Characterization techniques:

FTIR spectroscopy

- Equipment: Perkin Elmer Spectrum Two (USA).

XRD analysis:

- Equipment: Panalytical 3040/60 Xpert PRO (UK), using a CuKα source, with a maximum temperature of 80°C.

Surface morphology:

- Equipment: Jeol/EO CM version 1.1 (JSM 5910, Japan).

Thermogravimetric analysis (TGA):

- Equipment: Diamond TG/DTA (Perkin Elmer, USA) for evaluating thermal stability.

Dynamic mechanical properties:

- Equipment: DMA 242 C/1/G (Netzsch, Germany).
- Conditions: Compression mode, heating rate 5 °C/min, constant frequency 5 Hz, temperature range 0 to 100 °C (cooling with liquid nitrogen).

Tensile test:

- Equipment: Universal Testing Machine (UTM) Series 300 (Zwick Roell Z010, Germany).
- Sample size: 30 mm long, 5 mm wide; three samples were taken to ensure reliability.

Experimental conditions:

- Sample size: rectangular (6 mm x 5 mm).
- Testing conditions: Room temperature for tensile tests and controlled temperature for DMA analysis.

Hydrogel properties:

The roughness of the hydrogels was evaluated by the plot under the stress strain curve. A laboratory study used a bottle filled with 2.25 kg of cold drink (7-Up) to evaluate the load-bearing capacity of the hydrogel strips and observe the self-healing properties of the hydrogels.

Synthesis and properties:

- **Methods:** A novel method for the preparation of AG-based hydrogels was proposed using a one-step synthesis method using chemically unmodified acrylic acid (AA) and alginate (AG) as raw materials.
- **Interactions:** The hydrogels rely on physical interactions, including ionic electrostatic interactions between Fe^{3+} ions and carboxylate (COO^-) and hydroxyl (OH^-) groups in AA and AG. These interactions were confirmed by FTIR spectroscopy.
- **Additives:** XRD analysis verified the effective dispersion of TiO_2 particles, indicating that the mechanical properties of the hydrogels can be modified by adjusting the TiO_2 content.

Behavior and Applications:

- **Viscoelasticity:** Newtonian viscoelastic behavior was demonstrated by Cole-Cole plot analysis.

- **Applications:** The hydrogel has a long-term load-bearing capacity and rapid self-recovery ability, making it suitable for smart sensing devices, biomedical fields, and environmental research [58].

Conclusion

Biomaterials are highly regarded in the scientific community for their sustainability, biodegradability, and non-toxicity. Functionalization of these materials can modify their properties, making them competitive with traditional petrochemical products and expanding their research in specific application areas.

Initially, polyvinyl alcohol (PVA) was selected as the raw material for stabilizing polyvinyl acetate (PVAc) emulsions. However, starch has become a viable alternative for stabilizing PVAc emulsions. Like starch, cellulose and modified cellulose such as hydroxyethyl cellulose (HEC) belong to the polysaccharide family and are expected to play a similar role in emulsion stabilization.

The incorporation of these natural materials has shown an enhancing effect and helps reduce volatile organic compound (VOC) emissions by reducing the reliance on petrochemical additives. Notably, natural gums such as Arabic gum (AG) are promising candidates for use as protective colloids in PVAc polymerization. They offer several advantages, including renewable nature, biodegradability, and enhanced emulsion polymer stability and performance.

Abbreviations

HEC	Hydroxy ethyl cellulose
AG	Arabic gum
PVA	Polyvinyl alcohol
PVAc	Polyvinyl Acetate
VAc	Vinyl acetate monomer
VAM	Vinyl acetate monomer
Veova	Vinyl ester of Versatic acid
APS	Ammonium per sulphate
DH	Degree of hydrolysis
PSD	Particle size distribution
MFC	Micro fibrillated cellulose
MSC	Microcrystalline cellulose
CNF	Cellulose nanofibers
CNC	Cellulose nanocrystals
VOC	Volatile organic

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