Fabrication and Properties of polyacrylic acid by ionic surfactant disturbance method

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Abstract. The formation of polymeric materials can be achieved by several methods such as melting and casting, screw extrusion, cross-linking of resin or rubber in a mold, and so on. In this work, the polyacrylic acid is formed by using the emulsion disturbance method. Despite extensively used in the colour painting and coating industries, acrylic emulsion can be processed into a foam and powder configuration by a reaction between acrylic emulsion and salt. The solidification hardly changes the volume between liquid emulsion and solidified polymer which means the final structure of polyacrylic acid is filled with opened air cells. The opened air cell structure is confirmed by the result from scanning electron microscopy. The chemical analysis and crystallography of acrylic powder and foam are examined by Fourier-transform infrared spectroscopy and X-ray diffraction respectively. The phase transformation and Thermal stability are studied by differential scanning calorimetry and thermo gravimetric analysis. Moreover, the mechanical properties of acrylic foam were observed by tensile, compressive and hardness test. In addition to the basic property analysis, acrylic foam was also used in the particle filtration application.

1. Introduction

Nowadays, Acrylic emulsion is widely used in both academic and industrial due to its simplification in the fabrication process in which properties can be easily controlled [1]. Normally, acrylic emulsion is synthesized by a chemical process so called dispersion polymerization. This process utilizes the surfactant to control the degree of polymerization and the particle size including the soluability property. Due to its extensive applications in academic and industry, the copolymerizing process is added to improve the property of the acrylic. In general, the copolymer acrylic emulsion shows several good properties such as a prominent heat resistance, high water resistance, high corrosion resistance, stain resistance in glossiness and colour retention resistance [2]. Therefore these materials are widely used in building materials industry for example painting, coating materials and so on [3].

Polyelectrolytes are polymers with ionizable groups and soluble in polar solvents such as water [4]. The same as acrylic emulsion, polyelectrolytes are also widely used in many applications such as coatings, thin film and layer by layer [5]. The interest of these polymers are the reaction between

polyelectrolyte and salt which can be used to rapidly solidify the polyelectrolyte. Furthermore, the solidification of polyelectrolyte hardly changes the volume which make it suitable for casting in any designed mould [6]. On the other hand, in our case, the solidification of acrylic emulsion doesn't need the polyelectrolyte concept, but rather the ionic surfactant agglomeration. In term of property, although both polyelectrolyte and acrylic emulsion can be solidified by salt or ion charges, only polyelectrolyte is reversible between liquid and solid by water. The solidification of acrylic emulsion is permanent and can be used to fabricate a rigid material in any application.

In this work, acrylic powder and foam are formed by a reaction between acrylic emulsion and salt. Foam configuration was used in the particle filtration application. The effect of salt on structure and morphology of powder were investigated by FTIR spectroscopy, X-ray diffraction and SEM image. Phase transformation and thermal stability are studied by DSC and TGA. Tensile strength, hardness and compressive strength properties were used describe mechanical properties of foam.

2. Experiments

In this work, the raw material, which was acrylic emulsion, had a fixed concentration of 60% by weight. The main purpose of this experiment was to form the acrylic powder and foam by adding salt into acrylic emulsion. For acrylic powder, the fabrication can be divided into two methods. The first method was to add salt powder into the acrylic emulsion directly, and another method was to add the (20% by weight) salt solution into acrylic emulsion. In both case, the ratio between acrylic emulsion per salt (or salt solution) was 4:6 by weight. In this step, the mixture between acrylic emulsion and salt was automatically solidified. Later on, the sample was heat treated at 85 °C for 30 min in the air and dried in vacuum oven at room temperature for 12 hr. Finally, the sample was grinded into small powder by ball mill for 1 hr and then washed by water to remove salt. The morphology, chemical analysis and crystallography of acrylic powder are examined by scanning electron microscopy, Fourier-transform infrared spectroscopy and X-ray diffraction respectively. The phase transformation and thermal stability of acrylic powder are investigated by differential scanning calorimetry and thermo gravimetric analysis.

For acrylic foams, the fabrication method was the same as acrylic powder. The difference was that the ratio between acrylic emulsion and salt (or salt solution) was in a wide range between 8:2, 6:4, 4:6, and 2:8 by weight. The variation of the proportion was in order to observe the solidification property and the morphology of acrylic foam. After the foams were solidified, the heat treatment was performed in order to strengthen the acrylic foam. The mixtures were placed in the oven at 85 °C for 3 hr and washed in the water to remove salt. The final process was to dry the sample in vacuum oven at room temperature for 24 hr to obtained acrylic foam. The mechanical properties of acrylic foam were measured by tensile, compressive and hardness test. In addition to the basic property analysis, acrylic foam was also used in the micro-nano particle filtration application.

3. Results and discussion

3.1. Morphology and crystallography

By mixing salt in acrylic emulsion, the surfactant that encapsulated polymer in the emulsion was disturbed by the sodium ion and chloride ion. The result of the ion disturbance was the agglomeration of polymer chains in a defined container. We could immediately see the solidification or gelation of polymer soon after the salt was added into the acrylic emulsion. The morphology of acrylic powders was observed by scanning electron microscopy (SEM) as shown in Figure 1. Acrylic powder from NaCl powder and acrylic powder from NaCl solution were shown in Figure 1(a) and 1(b) respectively. The morphologies of acrylic powders from both NaCl powder and NaCl solution were rough which was different from the regular dispersive polymerization method [7]. The particle sizes of acrylic powders from NaCl powder and from NaCl solution were around 500 µm and 300 µm respectively. The size difference between two methods came from the kinematic of the reaction. Basically NaCl powder had a higher number of ions which can provide a stronger polymer agglomeration than NaCl solution.

Figure 1. SEM images of acrylic powder from NaCl powder (a) acrylic power from NaCl solution (b), XRD pattern of dried acrylic emulsion, acrylic powder from NaCl powder and acrylic powder from NaCl solution (b) and FTIR spectra of dried acrylic emulsion, acrylic powder from NaCl powder and acrylic powder from NaCl solution (d).

In Figure 1(c), XRD pattern from dried acrylic emulsion, acrylic powder from NaCl powder, and acrylic powder from NaCl solution demonstrated broad spectrums between 5° and 25°. This peak indicates that acrylic powder formed an amorphous phase [8]. FTIR result from the dried acrylic emulsion, acrylic powder from NaCl powder and acrylic powder from NaCl solution were shown in Figure 1(d) which corresponded to the OH stretching at 3441.06, CH asymmetric stretching at 2959.47, CH symmetric stretching at 2889.81 and C=O stretching at 1738.43 [9].

3.2. Phase transformation and thermal stability

The phase transformation and thermal stability of dried acrylic emulsion, acrylic powder from NaCl powder and acrylic powder from NaCl solution were observed by DSC and TGA as shown in Figure 2(a), (b) and (c) respectively. The TGA result showed the mass loss around 100 $^{\circ}$ C and 400 $^{\circ}$ C which complied with the decomposition temperature of water and polyacrylic acid respectively. These results indicated that acrylic powder from NaCl powder and acrylic powder from NaCl solution had the same phase transformation temperature and decomposition temperature as dried acrylic emulsion [10].

Figure 2. The phase transformation and thermal stability of dried acrylic emulsion, (a) acrylic powder from NaCl powder (b) and acrylic powder from NaCl solution (c). The observation temperature was between 25-900 °C and heating rate is 10 °C/min.

3.3. Mechanical properties

The mechanical properties of acrylic foam were characterized by tensile, hardness and compressive test. Tensile strength of acrylic foam were shown in Figure 3(a). Tensile strength of acrylic foam decreased with an increasing content of NaCl powder and solution, which reached 0.2 MPa at acrylic emulsion per NaCl powder ratio of 2:8, and 1.2 MPa at acrylic emulsion per NaCl powder ratio of 2:8. Hardness of acrylic foam was shown in Figure 3(b). The hardness of acrylic foam decreased with the increasing content of NaCl powder and solution. The compressive strength of acrylic foam was shown in Figure 3(c). The compressive strength of acrylic foam from NaCl powder decreased with the increasing content of salt powder. However compressive strength of acrylic foam from NaCl solution increased with the increasing content of salt solution. The overall mechanical properties of acrylic foam in this experiment were a bit lower compared to the strength of typical dried acrylic [11].

Figure 3. Mechanical properties of acrylic foam were observed by tensile, (a) hardness (b) and compressive test. (c).

3.4. Filtration application

Particle filtration of acrylic foam from NaCl powder was experimented by using the acrylic foam to filtrate ZnO nanorods. The filtrated water was observed by atomic absorption spectroscopy (AAS) and zeta sizer in Table 1. The result of filtration from AAS implied that the filtration efficiency was reduced with the increasing content of NaCl powder during the fabrication because the porosity size became bigger when the agglomeration from the ions is higher. The AAS result was coincided with the zeta sizer result since the number of ZnO nanorods that can pass through the filter also increased when the salt content had increased.

Table 1. Result of particle filtration application of acrylic foam from NaCl powder at difference NaCl powder content.

4. Conclusion

Acrylic powder and foam configuration are formed by using by a reaction between acrylic emulsion and salt. Morphology and crystallography were described by SEM image, XRD pattern and FTIR spectra. These result revealed that morphology of acrylic powder have rough surface and irregular shape. Acrylic powder had an amorphous phase and there was no chemical transformation in the acrylic bonding during the fabrication based on FTIR result. The overall mechanical properties were worsen if a higher content of salt was added. Result from particle filtration indicated that the filter can be used as a rough filtration for water treatment.

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