



NOTE

Syntheses of Series of Organosilicon-Acrylate Composite Emulsions for the Consolidation and Conservation of Historical Earthen Sites

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Since a couple of thousands of years ago, earth has been an economic construction material. Indeed, up to date, in many areas people still adopt earth to construct their houses. Ancient earthen buildings are heritages of all human beings. So, they must be preserved and delivered to our posterity. However, earthen buildings cannot resist the corrosion of wind and rain. For the sake of protecting the ancient earthen buildings, we synthesized a series of organosilicon-acrylate composite emulsions. These organosilicon-acrylate composite emulsions may effectively consolidate the historical earthen sites and allow them to oppose weathering.

Keywords: Composite emulsion, Consolidate, Earth, Heritage, Organosilicon-acrylate.

As a low-cost, universal and simple construction material, earth has been traditionally and widely used to build constructions for thousands of years. A large number of ancient earthen buildings have thus far been corroded by wind and rain and many collapsed, because earth is generally flimsy in wind and rain. In order to protect earthen buildings and turn over them to our next generations, steps need to be taken to consolidate and preserve these historical earthen sites. As a result, many people focus on the preparation of different protecting materials¹⁻⁵. Up to date, the protecting materials are generally grouped into two kinds, *i.e.* inorganic and organic materials. Inorganic materials like silicone resin and potassium silicate solutions are low-cost, aging resistance, little effect on earthen site appearance. However, they show large contraction and the reinforcement effect is not significant. Organic materials like acrylic resins are fine water and corrosion resistant, easy to combine with earthen wall, as well as have good reinforcement effect, but poor aging resistance. Considering the advantages of inorganic and organic materials, if combine both of them into a new material, it could has all of the advantages. Based on the above discussions, we design and prepare a series of organosilicon-acrylate composite emulsions which might protect earthen buildings against the corrosion of wind and rain. We report herein the preparation of these organosilicon-acrylate composite emulsions.

All the reagents were of A.R. grade and applied without further purification. The reagents include methyl acrylate, butyl

acrylate, ethyl acrylate, styrene, silane coupling agent KH570, silane coupling agent DL171, silane coupling agent DL602, complex surfactant SDS-OP-10 and Span80-Tween80, 2,2'-azobis(2-methylpropionitrile)(AIBN), benzoyl peroxide (BPO), *n*-pentanol, ammonium sulfate and sodium bisulfite. The apparatus are mainly electronic balance, vacuum pump, magnetic stirrer, water bath pot and three-necked round bottom flask.

Syntheses of organosilicon-acrylate composite emulsions

Methyl acrylate-styrene-KH570 composite emulsion: Methyl acrylate, styrene and silane coupling agent KH570 are the three elements of comonomer, with a mass ratio being of 25 : 25 : 2 g (weighed with an electronic balance). Putting 40 mL deionized water, complex surfactant SDS (2.5 g)-OP-10 (2.5 g) and *n*-pentanol (2.5 g) into a three-necked round bottom flask which was in a water bath pot, mixed evenly, then added 12.5 g methyl acrylate and 5 g styrene and stirred for about 1 h, forming a translucent emulsions. 100 mL 0.5 % potassium persulfate and sodium bisulfite aqueous solutions was then added into the translucent emulsions and slowly warmed up to 65 °C to carry out a emulsion polymerization reaction (when the system appearing blue fluorescence or temperature rise indicates that the reaction started) for 1.5 h, followed by slowly dropping 12.5 g methyl acrylate and 20 g styrene into the emulsion. As soon as the conversion ratio of the styrene being about 90 %, joined 2 g KH570 monomer rapidly and continued

to react for 1.5 h, then powered off the magnetic stirrer. Took out the three-necked round bottom flask from the water bath pot and cooled it down to 40 °C, then neutralized the emulsion to pH = 7 with 29 % ammonia solutions. Blue emulsion is obtained in a high yield by filtration.

Butyl acrylate-styrene-DL171 composite emulsion:

Butyl acrylate (25 g), styrene (25 g) and silane coupling agent DL171 (4 g) are the primary components of comonomer, with their mass weighed on an electronic balance. 83 mL deionized water, complex surfactant Span80 (2.7 g)-Tween80 (2.7 g) and *n*-pentanol (2.7 g) were moved into a three-necked round bottom flask which was soaked into a water bath pot, admixed them evenly with a magnetic stirrer, followed by dropping 12.5 g butyl acrylate and 5 g styrene and evenly stirring for around 1 h, yielding a translucent emulsions. 0.05 g 2,2'-azobis(2-methylpropionitrile) was put into the translucent emulsions, then slowly heated to 50 °C to bring a emulsion polymerization reaction (when the system showing pink fluorescence or temperature rise for about 3 °C indicates that the reaction has happened) for 1.5 h, followed by slowly adding the rest of butyl acrylate and styrene into the emulsion. 15 min. later, rapidly added 4 g DL171 monomer to the emulsion and continued to react for 1.5 h, then powered off the magnetic stirrer and removed the three-necked round bottom flask from the water bath pot. Cooled the three-necked round bottom flask with tap-water to 40 °C and neutralized the emulsion to pH = 7 with 29 % ammonia solutions. Pink emulsion is acquired by filtration.

Ethyl acrylate-styrene-DL602 composite emulsion:

By means of an electronic balance, we weighed the ethyl acrylate (25 g), styrene (25 g) and silane coupling agent DL602 (4 g) as the basic constituents of comonomer. Mixing 83 mL deionized water, complex surfactant Span80 (2.7 g)-Tween80 (2.7 g) and *n*-pentanol (2.7 g) into a three-necked round bottom flask that was dipped into a water bath pot, evenly mingled

them by virtue of a magnetic stirrer, then added 12.5 g ethyl acrylate and 5 g styrene and continued to evenly stir for 1 h, resulting in a translucent emulsions. 0.05 g of the initiator benzoyl peroxide was added into the translucent emulsions and slowly heated to 50 °C to cause a emulsion polymerization reaction (when the temperature rising for about 2-3 °C suggests that the reaction has occurred). 1.5 h later, slowly dropped the rest of ethyl acrylate and styrene into the emulsion and 15 min later, quickly added 4 g DL602 monomer to the emulsion and continued to react for another 1.5 h, then powered off the magnetic stirrer and removed the three-necked round bottom flask from the water bath pot. Cooled down the three-necked round bottom flask to 40 °C with tap-water and neutralized the emulsion to pH = 7 with 29 % ammonia solutions. White emulsion is obtained with filtration.

In conclusion, a series of organosilicon-acrylate composite emulsions were prepared, which might protect earthen buildings against the corrosion of wind and rain. Further studies on the properties of these organosilicon-acrylate composite emulsions will be carried out in our laboratory.

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