

# Cause Analysis of Soft Caking of VAE Emulsion

Jixue Lv<sup>1</sup>, Zhiwei Wang<sup>2\*</sup>

<sup>1</sup> Lianyungang Changrui Technology Co., Ltd., Lianyungang, Jiangsu, China

<sup>2</sup> Dalian Chemical Industry (Jiangsu) Co., Ltd., Yangzhou, Jiangsu, China

\*Corresponding Author.

## Abstract:

VAE emulsion is one of the copolymerization compounds of vinyl acetate and ethylene, and it is a widely used environmental protection product. VAE emulsion is mainly used in adhesives, coatings, textiles/non-woven fabrics, building cement, paper and carpets, etc. In this paper, the causes of soft caking during storage of VAE emulsion were discussed by simulation experiment. The formation mechanism of soft caking was confirmed by reducing the solid content of emulsion and adjusting pH value. The distribution of emulsion particles and soft caking was observed by electron microscope (SEM), and then the composition and components of soft caking were further determined. The glass transition temperatures of emulsion and soft caking were compared by DSC, and it was confirmed by experiments that the water loss of emulsion surface layer was the main reason for the formation of soft caking, which could provide reference for the application and storage of VAE emulsion.

**Keywords:** VAE emulsion, Soft caking.

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## I. PREFACE

VAE emulsion is one of the copolymerization compounds of vinyl acetate and ethylene, and it is a widely used environmental protection product. VAE emulsion is mainly used in adhesives, coatings, textiles/non-woven fabrics, building cement, paper and carpets, etc [1]. The output of VAE emulsion in China reached 750,000 tons/year in 2018. Because of the excellent performance of VAE emulsion, its annual output is increasing at a rapid rate of 20% [2]. At the same time, the progress of national environmental protection policies and standards has guided the development of adhesive products in an environment-friendly direction. Because of its low VOC, water-based VAE emulsion can be dried and formed into a film by itself, which is in line with the environment-friendly development direction. Its consumption is also increasing year by year [3]. A typical VAE emulsion is a white liquid formed by dispersing solid polymer particles in an aqueous medium. To prevent the polymer particles from sticking together and agglomerating again, there are some protective substances around the polymer particles [4]. The commonly used protective colloid is polyvinyl alcohol. To meet the application requirements of high-speed sizing fields (such as packaging, furniture, etc.), the solid content is increased to: at the same time, to improve the adhesion to low surface energy substances and increase the ethylene content, the higher the ethylene content in VAE emulsion, the lower the glass transition temperature and the smaller the surface tension of the emulsion (the softer the polymer film) [5]. To meet different application requirements, there are many kinds of VAE emulsion brands at present. During the application process, it

is found that there will be soft caking on the surface of VAE emulsion. However, there are few reports on the causes of soft caking of VAE emulsion. This paper focused on the simulation experiment to discuss the causes of soft caking during the storage of VAE emulsion, and confirmed the formation mechanism of soft caking by reducing the solid content of emulsion and adjusting the pH value. Electron microscope (SEM) was used to observe the distribution of emulsion particles and soft caking, and then the composition and components of soft caking were further determined. The glass transition temperatures of emulsion and soft caking were compared by DSC. It was confirmed by experiments that the water loss on the surface of emulsion was the main reason for the formation of soft caking, which could provide reference for the application and storage of VAE emulsion.

## II. MATERIALS AND METHODS

### 2.1 Simulation Experiment on the Cause of Soft Caking of VAE Emulsion

Four VAE products with different glass transition temperatures and different solid contents were selected. They were numbered A, B, C and D respectively. Their data are shown in Table I.

**TABLE I. Basic Data of VAE Emulsion**

Sample name	Solid content, wt%	Glass transition temperature (initial value), °C
A	55.8	9.58
B	60.6	0
C	55.8	-11
D	56.1	0

Add the above emulsion into half of a 20 mL sample bottle respectively, and bake them in a closed oven at 85°C for 14 hours. As shown in Figure 1.

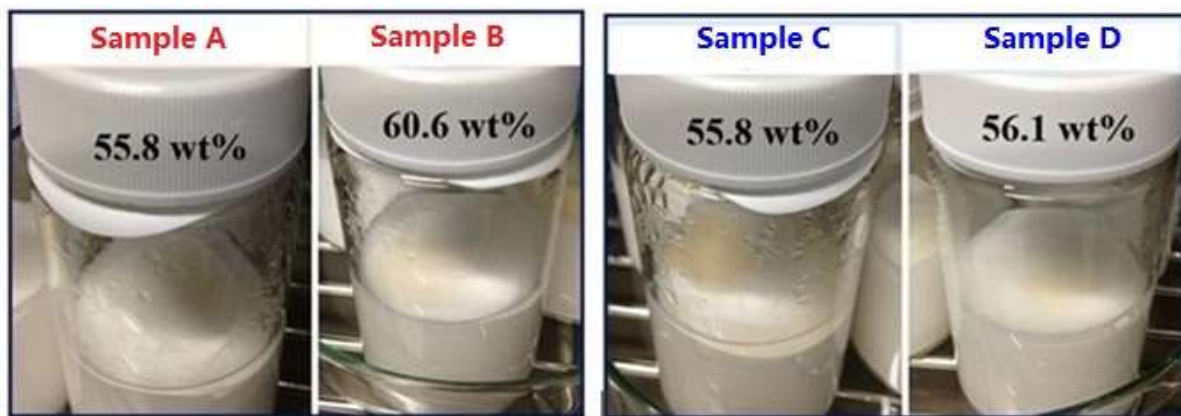


Fig 1: State of emulsion before soft caking test

After 14 hours of baking, it was found that all the emulsions had soft caking, which gathered in the center of the sample bottle and extended upward to the liquid level, as shown in Figure 2.



Fig 2: (a) Soft caking on the liquid surface of sample A and (b) sample B.

It is speculated that when the water in the emulsion evaporates at high temperature, the water vapor condenses on the bottle wall and flows down along the bottle wall. Because the temperature of the water flowing down is low, a flow field flowing towards the middle is formed in the solution, as shown in Figure 3.

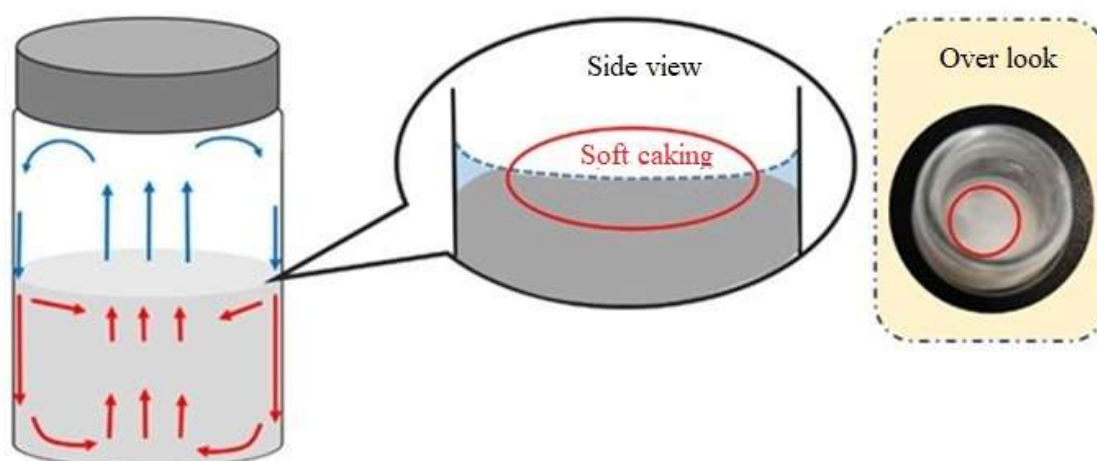


Fig 3: The mechanism of soft caking on the liquid surface.

When the viscosity decreases at high temperature, this flow field can drive the emulsion particles to move to the middle part and gather, resulting in soft caking extending to the liquid level.

## 2.2 Reducing the Solid Content of Emulsion

To improve the occurrence of soft caking on the liquid surface, we added a little water into the emulsion to reduce the solid content of the emulsion. Although aggregation still occurred, the solid content was not enough to aggregate on the liquid surface, and only settled below the liquid surface, which could solve the problem of soft caking on the liquid surface, as shown in Figure 4. It can be seen from Table II that when the solid content of the emulsion was reduced to about 50%, no soft caking would occur on the liquid surface.

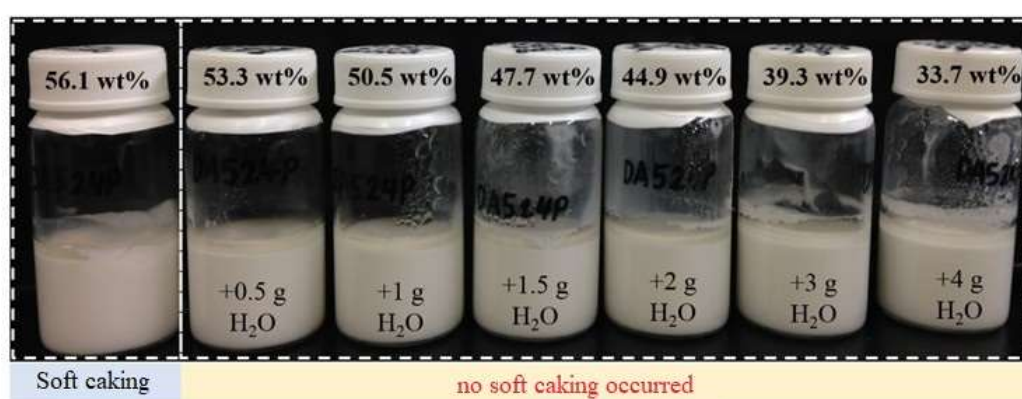


Fig 4: Sample D added with different water contents did not produce soft caking on the liquid surface

**Table II. Comparison table of soft caking on liquid surface caused by adding different water contents to various emulsions**

Sample	SC (wt %)	0.5g H <sub>2</sub> O+9.5 g Emulsion		1.0 g H <sub>2</sub> O+9.0 g Emulsion		2.0 g H <sub>2</sub> O+8.0 g Emulsion	
		Soft caking on liquid surface	SC (wt %)	Soft caking on liquid surface	SC (wt %)	Soft caking on liquid surface	SC (wt %)
Sample A	55.8	○	53.0	×	50.2	×	44.6
Sample B	60.6	○	57.6	×	54.5	×	48.4
Sample C	55.8	○	53.0	×	50.2	×	44.6
Sample D	56.1	×	53.3	×	50.5	×	44.9

- represents soft caking
- × represents no soft caking

### 2.3 Adjusting the pH Value of Emulsion

To further explore the causes of soft caking in emulsion, we used alkaline solution to adjust the pH value of emulsion. Because each emulsion was negatively charged and the redox potential value was between -34.38 and -7.85, we hope that the emulsion particles can be stably and uniformly dispersed by changing the charge distribution outside the emulsion particles. It can be seen from Table III that by adjusting the pH value to neutral with different alkaline solutions, soft caking will occur on the liquid surface after the soft caking test mentioned in 2.1. It can be seen that the pH value of emulsion cannot reduce the occurrence of soft caking.

**TABLE III. Comparison table of soft caking on liquid surface of various emulsions at different pH values**

Sample	pH	10 g Emulsion + NaOH solution		10 g Emulsion + NH <sub>3</sub> solution	
		pH	Soft caking on liquid surface	pH	Soft caking on liquid surface
Sample A	4.62	6.99	○	-	-
Sample B	4.83	7.02	○	-	-
Sample C	5.24	7.10	○	8.42	○
Sample D	5.08	6.84	○	8.42	○

### 2.4 Microscopic Observation of Emulsion and Soft Caking

Observe the distribution of emulsion particles and soft caking by electron microscope (SEM), and further determine the composition and components of soft caking as shown in Figure 5, in which sample A1 is the original emulsion, sample A2 is the soft caking of sample A1 collected for the first time, and sample A3 is the soft caking of sample A1 collected for the second time. Firstly, the soft caking was freeze-dried to freeze the state of the emulsion particles containing water, and the emulsion particles were successfully photographed. The particle size was close to that measured by particle size distribution instrument by about 1  $\mu$ m. Comparing the normal emulsion particles with the particles of emulsion with soft caking, it was found that there was no significant difference between emulsion with soft caking and normal emulsion under electron microscope.



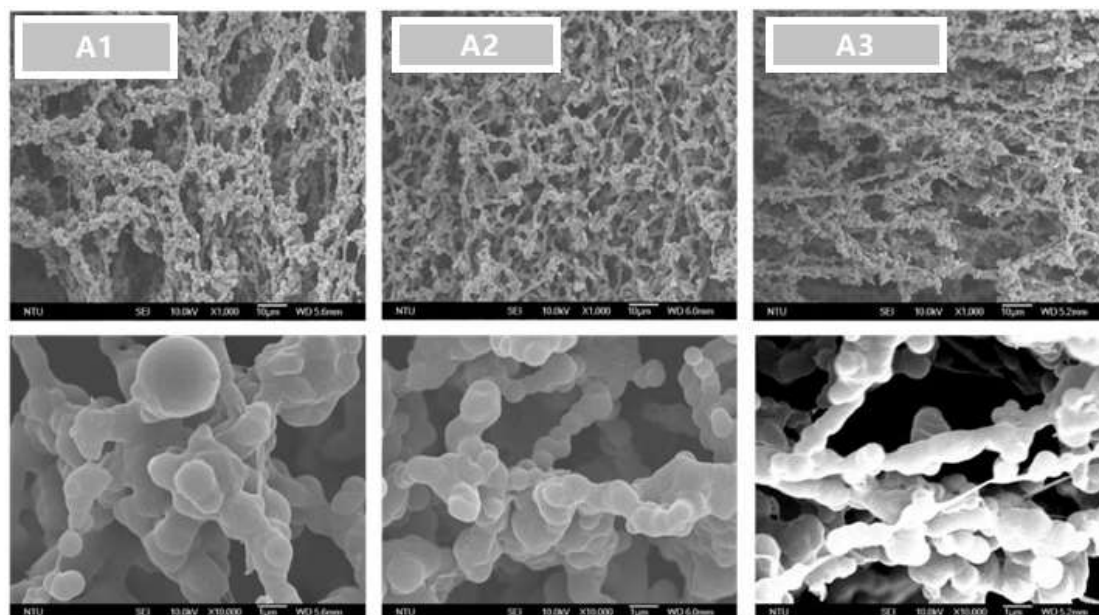


Fig 5: Observation of distribution of emulsion particles and soft caking by electron microscope (SEM)

## 2.5 DSC Measurement of Emulsion and Soft Caking

The soft caking A2 and A3 formed by normal sample A1 and A1 emulsion were measured by DSC [6]. The results are shown in Figure 6. It was found that there was no significant difference between the distribution of soft caking and normal samples, and the difference in glass transition temperature was too small to distinguish the difference. In all measurements, only the results of solid content were different. The solid content of normal emulsion was about 55wt%, and it was found that the solid content of all soft caking exceeded 60wt%. Among it, it was found that the harder A3 had 72wt% solid content. Based on the above experiments, it is speculated that the composition of soft caking should be the same as that of normal emulsion, but with less water content. This argument is consistent with the previous conclusion that water is the cause of soft caking.

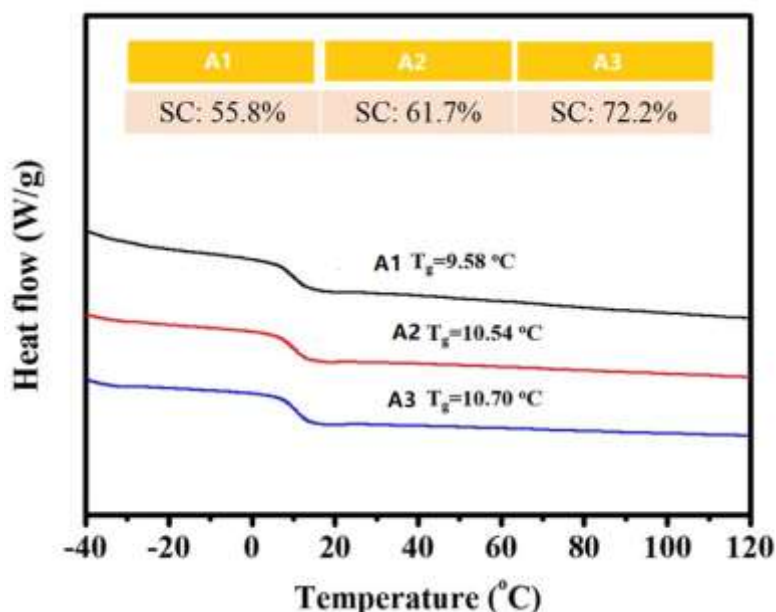


Fig 6: Thermal properties and solid content of sample A emulsion and its soft caking

### III. CONCLUSION

High viscosity is the main reason for the dispersion of VAE emulsion particles. When the temperature increases, the movement speed of emulsion particles increases, which will increase the chance of collision among particles and accelerate the aggregation phenomenon to lead to the occurrence of caking. Experience shows that caking often occurs at the liquid level. Under certain conditions, the surface water loss of emulsion is the main reason for the formation of soft caking. The formation of soft caking is mainly because of the loss of water. The solid content increases, while other factors such as emulsion particle distribution and glass transition temperature have not changed. The occurrence of soft caking quality problems of VAE emulsion can be effectively avoided only by taking necessary measures, such as humidification and sealing, in the process of filling, storage, transportation and use to avoid evaporation of water on the surface of the emulsion.

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