SUPPORTING INFORMATION

Effect of Carbon Number Distribution of Wax on the Yield Stress of Waxy Oil Gels

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1. HTGC. To determine the carbon number distribution of waxes, an Agilent 6890N high-temperature gas chromatograph with a capillary column HT750 (5 m × 0.53 mm × 0.09 μm) and a cool on-column inlet was used. For detection, a flame ionization detector (FID) was used and the detector temperature was 430 °C. The samples were dissolved in carbon disulfide before injection, and the solutions were injected into the column using an auto-sampler. The carrier gas used was helium, with a flow rate of 1 mL/min. The oven temperature was initiated at 40 °C and increased to 430 °C at a rate of 10 °C/min. Then temperature remained constant for 5 min. The solutions were run through the gas chromatograph several times, and an average composition was determined.

2. Determination of Experimental Conditions of Yield Stress Tests

2.1 Demonstration of no occurrence of setting. During the cooling process, the wax is precipitated from the oil when the temperature is below the WAT. It is likely that the wax particles may be settling down to the bottom of the vane rheometer. For the yield stress tests, it is necessary to ensure that the settling of wax particles does not occur. Due to the dependence of the setting on the particle size and time, the specimens of 12.5%W1+oil-A and 12.5%W3+oil-A were used to examine if the setting of wax crystals occurred during cooling and isothermal holding. The specimens at top and bottom of the rheometer were captured after the yield stress measurement. The WAT and wax content of samples were tested by using DSC, which will be presented in section 2.4 of the body of the paper. Table S1 summarizes the experimental results. As can be seen, the characteristics of the specimens from different positions of the rheometer are almost the same. In other words, setting of wax particles did not occur.

Table S1. WAT and wax content of specimens at top and bottom of the rheometer

Positions	12.5%W1+oil-A				12.5%W3+oil-A			
	CR=1 °C/min*		CR=0.5 °C/min		CR=1 °C/min		CR=0.5 °C/min	
	WAT (°C)	Wax content (wt %)	WAT (°C)	Wax content (wt %)	WAT (°C)	Wax content (wt %)	WAT (°C)	Wax content (wt %)
Top	38	23.4	38	24.0	46	24.8	46	24.2
Bottom	37	24.0	38	23.7	47	24.6	47	24.4

*CR is the abbreviation of cooling rate

2.2 Cooling Rate. The previous studies indicated that under quiescent condition, the yield stress increases with decrease of cooling rate due to formation of larger wax crystals at smaller cooling rate.[1, 2] The influence of cooling rate on the yield stress may be related to the carbon number distribution of wax. In this study, samples 12.5%W1+oil-A and 12.5%W3+oil-A were used to study the effect of cooling rate on the yield stress. The cooling rate of 0.5 and 1 °C/min was used, and the holding time was 100 min. Table S2 lists the yield stress results under different cooling rates. It was found that the yield stress increases as the cooling rate decreases, which is consistent with the results of previous studies.[1, 2] A cooling rate of 1 °C/min was finally used for all yield stress experiments to reduce the experimental time.

Table S2. Yield stress of wax-oil gels at 5 °C under different cooling rate

Cooling rate (°C/min)	Yield stress (kPa)	
	12.5%W1+oil-A	12.5%W3+oil-A
0.5	111.47	18.68
1	106.49	15.96

2.3 Holding Time. The yield stress also depends on the holding time. The effect of the holding time on the yield stress of samples 5%W1+oil-A, 5%W3+oil-A, 12.5%W1+oil-A, and 12.5%W3+oil-A was also studied. Figure S1 shows the variation of yield stress with the holding time. As can be seen, the yield stress of wax-oil gels approaches an asymptotic value after 100 min. Therefore, the holding time for yield stress tests in this study was determined to be 100 min.

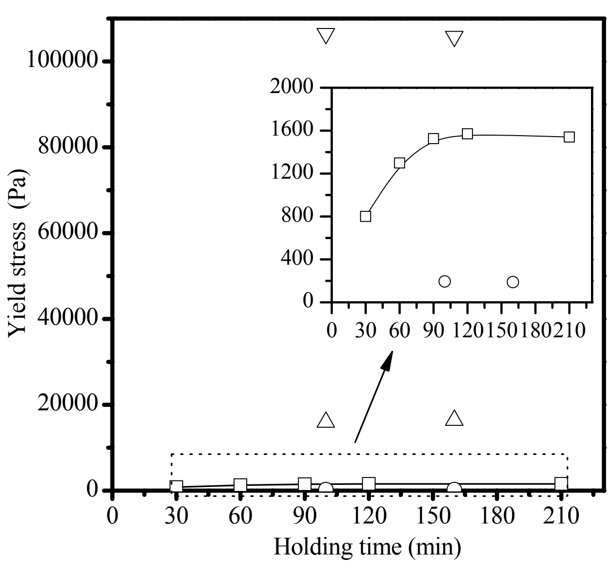

Figure S1. Yield stress vs holding time for samples 5%W1+oil-A (□), 5%W3+oil-A (○), 12.5%W1+oil-A (▽), and 12.5%W3+oil-A (△).

As will be presented in section 3.2 in the body of the paper, the small wax crystals in gels with high average carbon number of wax were observed, which results in small yield stress (cf. Figure 4). It is necessary to demonstrate that for the gels with high average carbon number of wax, small crystal size was not caused by the high degree of super-saturation and a significant crystallization at the holding period. To prove this, the sample 12.5%W3+oil-A was used, and the heat flow during the process of cooling and isothermal holding at 5 °C was measured by DSC. For the DSC measurement, the heating temperature and cooling rate were same with the conditions of yield stress tests.

Figure S2 shows the result of measurement of the heat flow. As can be seen, the thermogram does not show any detectable heat change during the isothermal holding at 5 °C. This can only occur in a system without occurrence of crystallization.[3] Therefore, it can be concluded that a significant crystallization of wax did not occur during the isothermal holding at 5 °C, and the small size of wax crystals in systems W3+oil-A was not because of the high degree of super-saturation at the holding period. In the previous published literature, it has been found that the structural features of the network formed by the wax crystals undergo change during the isothermal holding, especially during the initial holding time interval, so that the strength of the system increases with the holding time.[4] This may be the reason for the increase of yield stress with the initial holding time (cf. Figure S1).

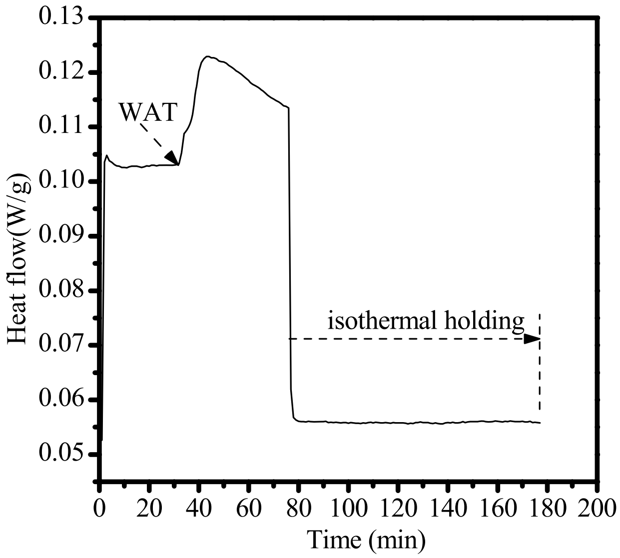

Figure S2. Variation of the heat flow for sample 12.5%W3+oil-A during the process of cooling and isothermal holding at 5 °C.

References

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