Preparation of High Encapsulation Efficiency Fragrance Microcapsules and Application in Textile

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Encapsulation efficiency

A certain amount of core materials (DPO/GTCC = 1:1) was dissolved into toluene and a series of standard samples (1.55, 3.10, 4.66, 6.21 and 9.31 mg/ml) were prepared. Firstly, one of the standard samples was scanned via UV visible spectrophotometer (Lamda 35, PerkinElmer) over the range of 200-1100 nm and the maximum absorbance of the core materials was 283 nm shown in **Fig.S1**. And then, the UV absorbances of the five standard samples were tested at 283 nm, respectively. The linear relationship between the concentration of core materials (x) of the five standard samples and their UV absorbance (y) was created according to the calibration curve of spectrophotometry; the equation is y=0.00249+0.08915x shown in **Fig.S2**. Finally, microcapsule slurry (3 g) of each sample in the **Table 1** was added to toluene (29 ml) with magnetic stirring for 30 seconds, respectively. The unencapsulated core materials (free oil) were extracted from the microcapsule suspension into the toluene. The supernatant solution (toluene solution) was separated from the microcapsule suspension. Then the supernatant solution was put into a cuvette for testing. And the UV absorbance (y) of the free oil solution was tested by UV visible spectrophotometer. The concentration of free oil solution (x) was calculated through the linear relationship, leading to the whole free oil (W_{FO}) of the sample. After that, the encapsulation efficiency (EE) of the core materials was determined by the following equation (1).

Encapsulation efficiency (EE) = $1 - W_{FO}/W$ (1)

 W_{FO} is the amount of free oil in the sample suspension and W is the amount of feeding core materials.



Fig.S1 The UV-Vis absorbance of core materials



Fig.S2 The relationship of UV-Vis absorbance with concentration of free oil

Washing durability and oil loading capacity

A certain amount of core materials (DPO/GTCC = 1:1) was dissolved into ethanol and a series of standard samples (0.047, 0.118, 0.237, 0.355 and 0.474 mg/ml) were

prepared. Firstly, one of the standard samples was scanned via UV visible spectrophotometer (Lamda 35, PerkinElmer) over the range of 200-1100 nm and the maximum absorbance of the core materials was 248 nm. And then, the UV absorbances of the five standard samples were tested at 248 nm, respectively. The linear relationship between the concentration of core materials (x) of the five standard samples and their UV absorbance (y) was created according to the calibration curve of spectrophotometry; the equation is y=1.74563x-0.00618 shown in **Fig.S3**. The fabric samples were impregnated in ethanol solution for 24 h. The DPO inside the fragrance microcapsules was extracted through the shell material into the ethanol solution. After that, the fabric samples were separated from the ethanol suspension. The supernatant solution was put into a cuvette to test the concentration of DPO, which contributed to calculate the concentration of DPO in the initial fabric sample. Finally, the mass of DPO in the sample was attained.



Fig.S3 The relationship of UV-Vis absorbance with concentration of core materials

Influence of curing temperature on encapsulation efficiency

The influence of curing temperature on EE was shown in the **Fig.S4**. The corresponding samples were from A_1 to A_4 in the **Table S1**. With the curing temperature increasing from 30 °C rpm to 60 °C rpm, the EE decreased first and then increased as shown in the **Fig.S4**. When the curing temperature was 40 °C, the EE was relatively maximum (85.6 %). In conclusion, the best homogenization rate was

40 °C based on EE.



Fig.S4 Effect of curing temperature on the encapsulation efficiency

Influence of homogenization time on encapsulation efficiency

When the curing temperature was 40 °C, the EE was variable with the homogenization time increasing, which was shown in **Fig.S5**. The corresponding samples were given from B_1 to B_5 in the **Table S1**. As illuminated in **Fig.S5**, the EE increased first and then decreased with the homogenization time increasing from 2 to 6 minutes. EE was the relatively maximum (86.42 %) when the homogenization time was 4 minutes. In conclusion, 4 minutes was the best homogenization time based on EE.



Fig.S5 Effect of homogenization time on the encapsulation efficiency

Influence of the ratio of core/shell on encapsulation efficiency

The ratio of core/shell had a significant effect on EE, as shown in **Fig.S6**. The specific change of ratio of core/shell was shown from C_1 to C_5 in the **Table S1**. As shown in **Fig.S6**, with the ratio of core/shell increasing, EE rapidly increased first and then decreased slowly. Specifically, as the ratio of core/shell from 2 to 6, EE rapidly increased. When the ratio of core/shell was greater than 6, EE began to decrease. Accordingly, the best ratio of core/shell was 6 based on EE.



Fig.S6 Effect of the ratio of core/shell on the encapsulation efficiency

Influence of homogenization rate on encapsulation efficiency

The influence of homogenization rate on EE of the microcapsules was shown in the **Fig.S7**. The corresponding samples were from D_1 to D_5 in the **Table S1**. With the homogenization rate increasing from 5000 rpm to 9000 rpm, the EE decreased first and then increased as shown in the **Fig.S7**. When the homogenization rate was 7000 rpm, EE was the relatively maximum (89.2 %). In conclusion, 7000 rpm was the best homogenization rate based on EE.



Fig.S7 Effect of homogenization rate on the encapsulation efficiency

experimental conditions				
	Curing temperature (°C)	Homogenization time (min)	Ratio of core/shell	Homogenization rate (rpm)
A_1	30			
A_2	40	2	6	6000
A_3	50	5	0	0000
A_4	60			
B ₁		2		
B_2		3		
B_3	40	4	6	6000
B_4		5		
B_5		6		
<u> </u>			2	
C_2			4	
$\overline{C_3}$	40	4	6	6000
C ₄			8	
C_5			10	
$\frac{1}{D_1}$				5000
D_2				6000
$\tilde{D_3}$	40	4	6	7000
\mathbf{D}_{4}	-		-	8000
D_5				9000

 Table S1 Feed mass of each component for preparation of microcapsules and

experimental conditions

Influence of homogenization rate on the sizes

The influence of homogenization rate on the sizes of the microcapsules was shown in

the **Fig.S8**. With the homogenization rate increasing from 5000 rpm to 9000 rpm, the size decreased first and then increased. When the homogenization rate was 7000 rpm, the size was the relatively minimum.



Fig.S8 Effect of homogenization rate on the sizes