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Taguchi method minimizes cost & time for determining optimum value of process parameters

Synthesis & study of microcapsules with beeswax core and phenol-formaldehyde shell by Taguchi method

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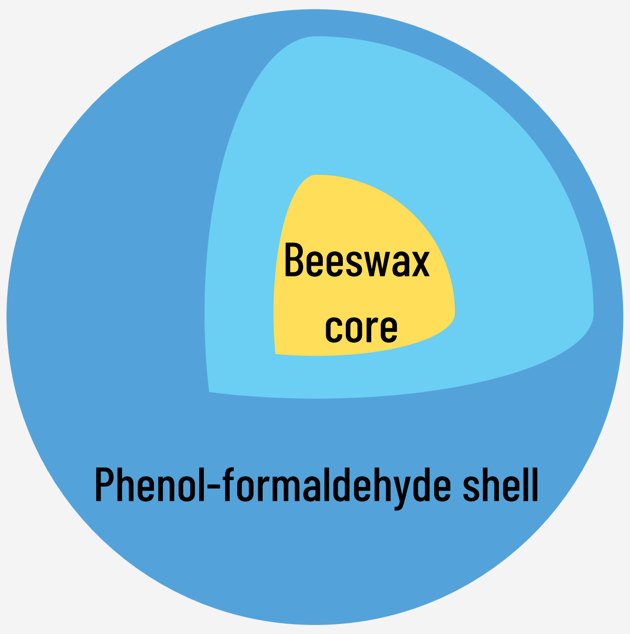

Figure S1: Core-shell morphology

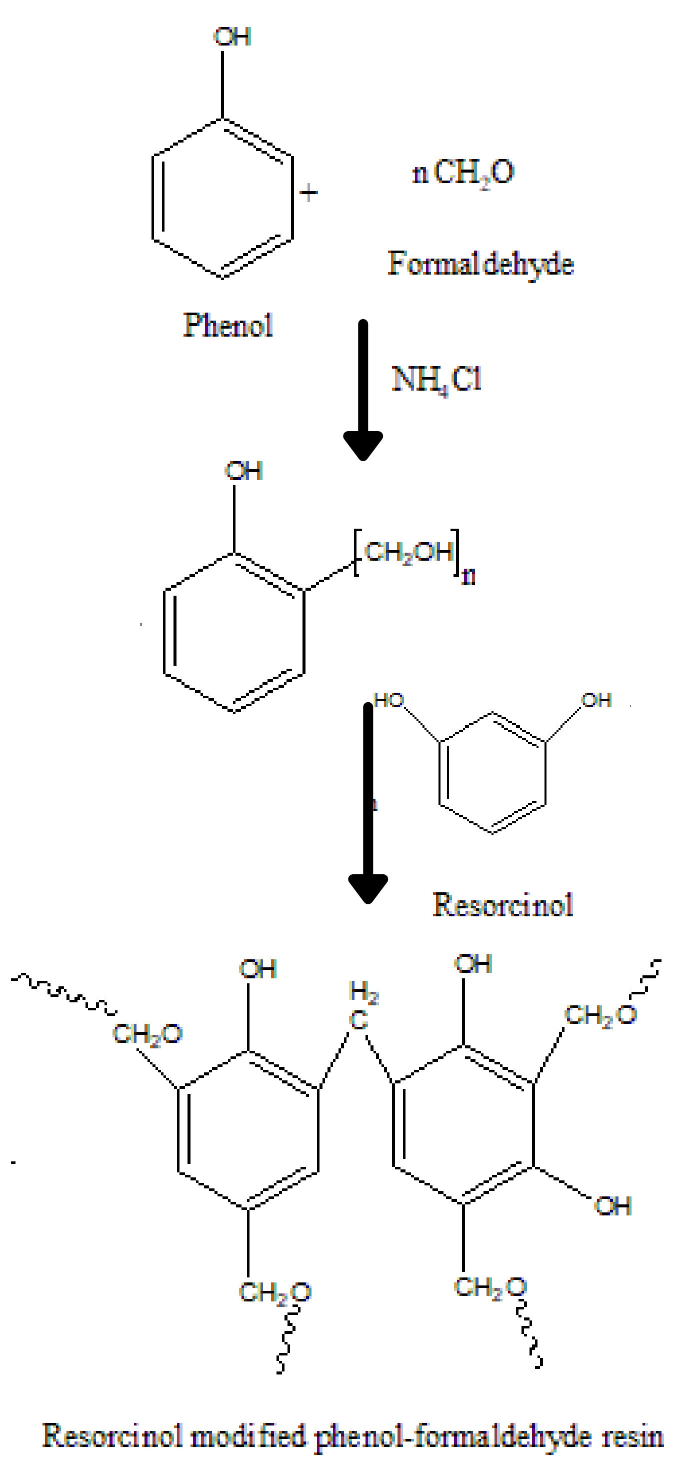

Figure S2: Polymerization reaction

INTRODUCTION

Bioproduct beeswax has good potential to serve as phase change material (PCM) for thermal energy storage applications. PCMs can absorb, store or release latent heat while undergoing phase transition at predetermined temperature range. When PCM is combined with other substrate, PCM melts from crystalline phase while temperature of the substrate is prevented from rising. Similarly, during crystallization process from molten phase, cooling of substrate is prevented. This phenomenon maintains the required application temperature in PCM incorporated substrate. But leakage problem during thermal transition process of PCM demands for encapsulation. In this study, beeswax was encapsulated by resorcinol modified phenol-formaldehyde shell to yield structure as shown in figure 1. The parameters varied were core/shell ratio, surfactant concentration & agitation speed.

OBJECTIVE

To obtain optimum values of process parameters.

Table S1: Taguchi L25 orthogonal array

Run [No]	Surfactant concentration [g]	Core/shell ratio [moles]	Agitation speed [rpm]	Core content (%)	SNR [dB]
1	1	0.5:1	400	61.2	35.75
2	1	01:01	600	72	37.14
3	1	1.5:1	800	70.2	36.92
4	1	02:01	1000	50.4	34.04
5	1	2.5:1	1200	19.8	25.93
6	2	0.5:1	400	76.8	37.70
7	2	01:01	600	77.4	37.77
8	2	1.5:1	800	66.6	36.46
9	2	02:01	1000	54	34.64
10	2	2.5:1	1200	77.4	37.77
11	3	0.5:1	400	73.8	37.36
12	3	01:01	600	78	37.84
13	3	1.5:1	800	67.5	36.58
14	3	02:01	1000	73.5	37.32
15	3	2.5:1	1200	70.5	36.96
16	4	0.5:1	400	72.15	37.16
17	4	01:01	600	77.4	37.77
18	4	1.5:1	800	62.4	35.90
19	4	02:01	1000	60	35.56
20	4	2.5:1	1200	69.6	36.85
21	5	0.5:1	400	48	33.62
22	5	01:01	600	61.5	35.77
23	5	1.5:1	800	76.8	37.70
24	5	02:01	1000	63.6	36.06
25	5	2.5:1	1200	60	35.56

METHODOLOGY

Microcapsules prepared by suspension polymerization with PVA surfactant. 2.1 g phenol and 0.5g ammonium chloride were dissolved in PVA-water solution for 30 minutes. At pH 7, beeswax-xylene solution ultrasonicated at 60 ºC for 5 minutes. This solution was added to 3.35 g of formaldehyde solution and heated to 65 ºC under stirring at 500 rpm for 2 hours. At pH 3, 0.5 g of resorcinol was added. Reaction was continued for 2.5 hours. Reaction can be studied with figure 2. Obtained microcapsules were vacuum filtered, washed & dried. Core content of microcapsules was calculated for different levels of parameters as shown in table 1. Larger the better approach was used.

ANALYSIS

The effect of parameter values on core content can be studied with main effects plots of SN ratio which is shown in figure 3. Increasing surfactant concentration gave finer emulsion with better dispersion. As the surfactant concentration increases above 3 wt.%, core content reduces. This is the reason for decrease in SNR value. Increase in core content was observed for core to shell ratio 1:1 and 0.5:1. Further increase in the ratio reduced shell thickness. The ruptured thin shell can show low core content. Increase in speed up to 800 rpm help in formation of core/shell morphology, Increasing the speed above this may rupture the shell. So, the optimized value for surfactant concentration, core to shell ratio and agitation speed were 3%, 1:1 and 800 rpm. The size and melting enthalpy of encapsulated PCM with optimized parameters was 62.61 µm and 148.93J/g in the range of 35-62 ºC respectively.

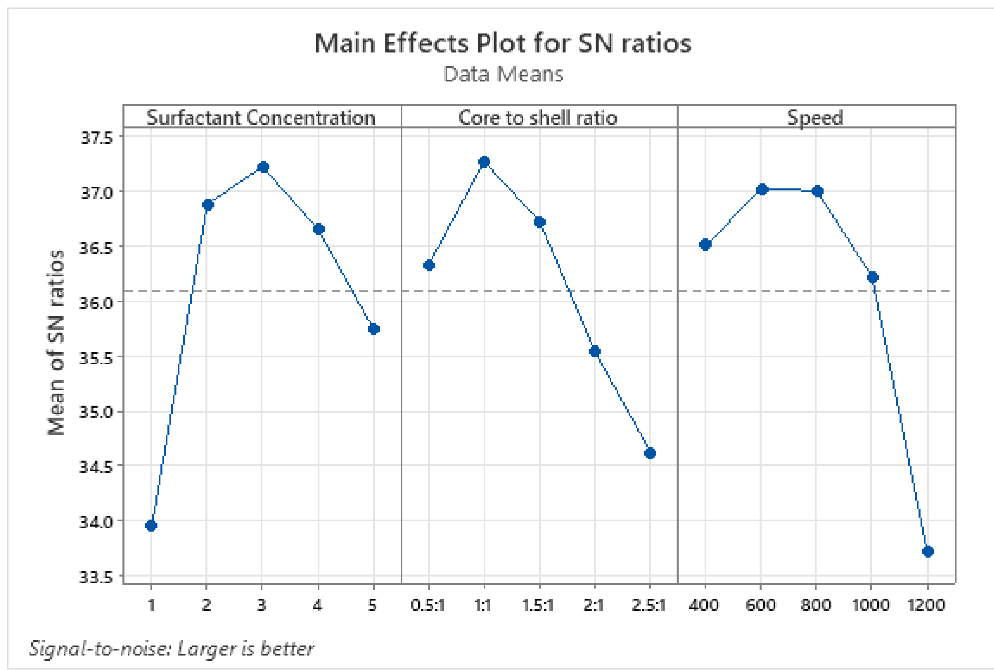

Figure S3: Main effects plots of SN ratio

CONCLUSION

The effect of surfactant concentration, core to shell ratio and agitation speed on core content of encapsulated PCM was studied. The optimized value for surfactant concentration, core to shell ratio and agitation speed were 3%, 1:1 and 800 rpm. The melting enthalpy, small size and temperature range of phase transition are suitable for thermal energy storing applications.