Optimization of technological parameters for preparation of lycopene microcapsules

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Abstract Lycopene belongs to the carotenoid family with high degree of unsaturation and all-trans form. Lycopene is easy to isomerize and auto oxide by heat, light, oxygen and different food matrices. With an increasing understanding of the health benefit of lycopene, to enhance stability and bio-availability of lycopene, ultrasonic emulsification was used to prepare lycopene microcapsules in this article. The results optimized by response surface methodology (RSM) for microcapsules consisted of four major steps: (1) 0.54 g glycerin monostearate was fully dissolved in 5 mL ethyl acetate and then added 0.02 g lycopene to form an organic phase, 100.7 mL distilled water which dissolved 0.61 g synperonic pe(R)/F68 as the aqueous phase; (2) the organic phase was pulled into the aqueous phase under stirring at 60 °C water bath for 5 min; (3) the mixture was then ultrasonic homogenized at 380 W for 20 min to form a homogenous emulsion; (4) the resulting emulsion was rotary evaporated at 50 °C water bath for 10 min under a pressure of 20 MPa. Encapsulation efficiency (*EE*) of lycopene microcapsules under the optimized conditions approached to 64.4%.

Keywords Lycopene · Ultrasonic emulsification · Microencapsulation · Response surface methodology

Introduction

Lycopene is a red carotenoid pigment and phytochemical found in red fruits and vegetables, such as tomatoes, red

carrots, watermelons and papayas. More and more literatures implicate that supplementation of food rich in lycopene is associated with the decrease of risk of cardiovascular diseases and cancers (Rao and Agarwal 1999; Kim et al. 2011). So lycopene has important and significant commercial value (Mysore Doddaiiah et al. 2011; Gunjan et al. 2009). The high degree of unsaturation in lycopene structure renders it extremely sensitive to light, oxygen and moisture (Macías-Sánchez et al. 2005; Lee and Chen 2002; Mascio et al. 1989).

Conventional methods to prepare of lycopene microcapsules rely on spray-drying (Koç et al. 2011; Sagar and Suresh Kumar 2010; Yousefi et al. 2011), phase separation, co-crystallization, interfacial polymerization (Catherine 2009) and microwave-assisted (Abbasi 2008) etc. Spray-drying is a popular way to encapsulate raw material and produce powder with good flowing property and water solubility (Oliveira et al. 2009), but the products are unsatisfactory. For example, Shu (2006) prepares lycopene microcapsules by spray-drying method using a wall system consisting of gelatin and sucrose. Lycopene has isomerization and the storage stability of products is poor at room temperature. Blanch et al. (2007) prepares lycopene microcapsules by supercritical fluid extraction method using α -, β - and γ -cyclodextrins (CDs) as wall materials. The use of supercritical fluids in food is highly recommended, because it avoids the employment of large amounts of organic solvents. But the costs of equipment and operation are so high and the technological parameters need further research.

Ultrasonic emulsification, as a better alternative with lower energy consumption and using of less surfactant, is gaining interest in many industrial fields comparing to other mechanical process. Although ultrasonic waves possess a high potential for liquid stirring and mixing, few studies have focused on using this technique to prepare lycopene

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microcapsules. To enhance the water solubility and storage stability of lycopene in this article, ultrasonic emulsification technics optimized by RSM experiment (Jain et al. 2011; Guo et al. 2007; Li et al. 2008; Liang et al. 2008) was studied to obtain the lycopene microcapsules.

Materials and methods

Materials Lycopene was purchased from Xinchang Pharmaceutical Factory (Zhejiang, China); glyceryl monostearate was purchased from Hangzhou shuoye oil chemical plant (Zhejiang, China); synperonic pe(R)/F68 was purchased from BASF Aktiengesellschaft 67056 Ludwigshafen (Germany); all solvents and reagents were of analytical grade.

Equipments SCIENT2-ID Supersonic Wave Comminution of Cell Equipment (Ningbo Xinzhi Siological Technology Ltd. China); Ultraviolet Visible Spectrophotometer (model 752) (Shanghai Spectrum Equipment Ltd. China); U570-ultra Low Temperature Freezer (New Brunswick Scientific, UK Ltd.); Christ ALPHA 2–4 LSC Freeze Dryer (Martin Christ, Germany); Mastersizer 2000 Laser Particle Size Analyzer (UK); CX21FS1 Optical Biomicroscopy (Olympus, Japan); Differential Scanning Calorimeter (DSC) (model 910) (DuPont Co. UK).

Selection of microcapsules systems The blank microcapsules systems were studied with different kinds of solvent systems (ethyl acetate, n-hexane, acetone, petroleum ether and dichloromethane), surfactants (Tween-80, synperonic pe(R)/F68, cremophor RH40, sodium alginate, polyvinyl alcohol, sucrose fatty acid esters, lecithin, gelatin and arabic gum) and carrier materials (stearic acid, glycerin monostearate and palmitic acid) in accordance with their stability constant of microcapsules (Cui 2004a, b), and the stability constant was calculated as:

$$K_e = \frac{A_0 - A}{A} \times 100\% \quad (1)$$

K_e stability constant

A_0 the absorbance of emulsion before centrifugation

A the absorbance of emulsion after centrifugation at 4000 r/min for 15 min

Preparation of lycopene microcapsules Specific mass of glycerin monostearate ($M_{\text{glycerin monostearate}}$) was fully dissolved in 5 mL ethyl acetate and then add 0.02 g lycopene to form an organic phase, the distilled water dissolved specific mass of synperonic pe(R)/F68 as the aqueous phase. Organic phase was pulled into the aqueous phase under stirring at 60 °C water bath for several min (T_{mixed}). Then, the mixture was ultrasonic homogenized in ultrasonic comminution of cell equipment for several min ($T_{\text{ultrasonic}}$) to form a homogeneous emulsion under a certain homogenization pressure ($P_{\text{ultrasonic}}$). Effects of $V_{\text{aqueous}}/V_{\text{oil}}$ (5 to 25), $M_{\text{synperonic pe(R)/F68}}$ (0.20 to 1.00 g), $M_{\text{glycerin monostearate}}$ (0.30 to 0.70 g), T_{mixed} (1 to 5 min), $P_{\text{ultrasonic}}$ (190 to 570 W) and $T_{\text{ultrasonic}}$ (10 to 30 min) on encapsulation efficiency were evaluated, respectively. All the analyses were conducted three times under the same experimental conditions.

Analysis of encapsulation efficiency The EE was calculated as the ratio between the initial mass of lycopene to be encapsulated and its mass in the final product. About 10 mL lycopene microcapsule homogeneous solution was decolored by active carbon, and then the encapsulated lycopene was extracted by dichloromethane and dried by 30 g anhydrous Mg_2SO_4 . The absorbance value of lycopene can be measured at 514 nm by Ultraviolet Visible (UV) spectrophotometry (Shu 2006). The standard curve was a straight line by UV analysis and its regressive equation Eq. 2 was obtained. Then, the concentration of lycopene can be calculated by it, and the total mass of lycopene can be obtained. Meanwhile, the EE of lycopene microcapsule was calculated via Eq. 3.

$$C = \frac{(A + 0.003)}{0.1777} \quad (2)$$

C the concentration of lycopene
 A the absorbance value of lycopene
 Linear range 0.8~4.0 $\mu\text{g}\cdot\text{mL}^{-1}$ $r=0.9999$

$$EE = \frac{M_1}{M_0} \times 100\% \quad (3)$$

EE encapsulation efficiency (%)

M_0 the initial mass of lycopene

M_1 the encapsulated mass of lycopene

Table 1 Effect of the different surfactants on stability constant (K_e)

Surfactants	Tween-80	Synperonic pe(R)/F68	Cremophor RH40	Sodium alginate	Polyvinyl alcohol	Sucrose fatty acid esters	Gelatin	Arabic gum
K_e [%]	135.7±4.37	4.4±0.13	3.5±0.08	32.5±1.34	16.7±0.75	60.0±1.78	42.9±1.97	894.3±9.94

$n=3$ panelists ($p<0.05$)

Table 2 Effect of the different carrier materials on stability constant (Ke)

Carriers	Stearic acid	Glyceryl monostearate	Palmitic acid
Synperonic pe(R)/F68, Ke [%]	135.0±3.64	3.5±0.07	208.3±5.69
Cremophor RH40, Ke [%]	105.4±1.56	5.7±0.12	56.8±1.85

$n=3$ panelists ($p<0.05$)

Optimization significant variables by response surface methodology (RSM) The Plackett-Burman (PB) design (Plackett and Burman 1946; Chakraborty et al. 2011) factorial designs allow for the screening of main factors from a large number of process variables. A total of six variables were analyzed with regard to their effects on the EE of lycopene microcapsules using a PB design. Each independent variable was tested at high (+1) and low (−1) levels. The software Design Expert 7.1 was used for experimental design, data analysis and quadratic model building. Then, not only find the vital factors, but also discover the optimal product formulations.

The significant parameters selected by PB design were optimized by RSM. The experimental results were fitted via the response surface regression procedure, using the following second order polynomial equation:

$$EE = \beta_0 + \sum_i \beta_i X_i + \sum_{ii} \beta_{ii} X_i^2 + \sum_{ij} \beta_{ij} X_i X_j \quad (4)$$

In which, EE is encapsulation efficiency (%), EE_i is the predicted response, $X_i X_j$ is independent variables, β_0 is the offset term, β_i is the i -th linear coefficient, β_{ii} is the ii -th quadratic coefficient, and β_{ij} is the ij -th interaction coefficient.

Preparation of freeze drying sample Lycopene microcapsules were prepared by freeze-dried (Dhewa et al. 2011; Sharma et al. 2011) 24 h at −70 °C and then freeze drying for another 24 h, 5% mannitol added as the protective agent.

Research on the stability of the products About 13.0 g freeze-dried lycopene microcapsules were kept at 4 °C for 2 months. Every 10 days, 1.5 g freeze-dried lycopene microcapsules were added 10 mL water to form homogeneous solution. Then, it was decolored by 20 g active carbon, filtered through qualitative filter paper, and the encapsulated lycopene in filtrate was extracted by dichloromethane and dried by 30 g anhydrous Mg_2SO_4 . The concentration of lycopene can be calculated via the Eq. 2, and the retention percentage of lycopene in microcapsules is calculated via the following equation:

$$RP = \frac{W_i}{W_0} \times 100\% \quad (5)$$

RP the retention percentage of lycopene in microcapsules (%)

W_0 the initial mass of lycopene in microcapsules

W_i the i -th mass of lycopene in microcapsules

The thermal stability of freeze-dried samples of lycopene microcapsule was studied by differential scanning calorimetry (DSC) (Nopianti et al. 2011).

Statistical analysis The results were expressed as means ±SD of triplicate measurements. Data were analyzed using the analysis of variance (ANOVA) technique while followed by Student's t -test ($p<0.05$) using SAS software (version 9.1 for windows). All statistical evaluations of RSM were done with the statistical software Design Expert 7.1.

Results and discussion

Selection of microcapsules systems Since the solubility and edible security of food industry, ethyl acetate was selected as solvent system to prepare lycopene microcapsules. Compared with the Ke value of different surfactants, the synperonic pe(R)/F68 and cremophor RH40 were the more relatively appropriate surfactant for the microencapsulation

Table 3 Experimental design by Plackett–Burman design for screening of reagent combination affecting encapsulation efficiency

Run	X_1	X_2	X_3	X_4	X_5	X_6	EE ^b
1	−1	−1	1	1	1	−1	33.1±1.25
2	−1	1	1	−1	−1	−1	22.4±0.94
3	−1	1	−1	1	−1	1	21.9±0.83
4	−1	−1	−1	1	1	1	24.3±1.02
5	1	−1	1	1	−1	1	33.9±1.41
6	1	1	1	1	1	−1	38.8±1.55
7	1	1	−1	−1	1	1	28.7±1.13
8	1	1	−1	1	−1	−1	22.1±0.74
9	1	−1	1	−1	−1	1	25.6±0.82
10	−1	−1	−1	−1	−1	−1	21.5±0.66
11	1	−1	−1	−1	1	−1	31.5±1.35
12	−1	1	1	−1	1	1	27.8±1.19

^a X_1 - $V_{a/o}$ (mL); X_2 - $T_{ultrasonic}$ (min); X_3 - $M_{synperonic\ pe(R)/F68}$ (g); X_4 - $P_{ultrasonic}$ (W); X_5 - $M_{glycerin\ monostearate}$ (g); X_6 - T_{mixed} (min); X_7 , X_8 , X_9 , X_{10} , X_{11} were blanks.

^b EE-encapsulation efficiency(%) ($p<0.05$) ($n=3$)

Table 4 Analysis of variance for selected factorial model

Source	Sum of square	DF	Mean square	F-value	P-value Prob > F
Model	268.8	3	89.6	8.5	0.007
X_1 - $V_{\text{water/oil}}$ (mL)	73.0	1	73.0	6.9	0.030
X_3 - $M_{\text{synperonic pe(R)/F68}}$ (g)	82.3	1	82.3	7.8	0.023
X_5 - $M_{\text{glycerin monostearate}}$ (g)	113.5	1	113.5	10.8	0.011
Residual	84.2	8	10.5		
Cor Total	353.0	11			

system (Table 1). The glycerin monostearate was the optimal carrier material for the microencapsulation system, and the synperonic pe(R)/F68 was the optimal surfactant (Table 2). So the blank microcapsules systems using synperonic pe(R)/F68 as surfactant, glycerin monostearate as carrier material, and ethyl acetate as organic solvent, were most stable.

Research on preparation of lycopene microcapsules by single factor experiments Several factors affecting EE of lycopene microcapsules were investigated in details. The formation of lycopene microcapsules was: 0.55 g glycerin monostearate was fully dissolved in 5.00 mL ethyl acetate and then added 0.02 g lycopene to form an organic phase; 0.60 g synperonic pe(R)/F68 was dispersed in 101.0 mL distilled water as the aqueous phase; organic phase was pulled into the aqueous phase with agitation in 60 °C water bath for 5 min; the mixture was ultrasonic homogenized in an ultrasonic comminution of cell equipment under 380 W for 20 min to form a homogenous emulsion, then which was rotary evaporated in a pear-shaped flask at 50 °C and 20 MPa.

Selection of significant variables by PB design For the selection of significant variables for lycopene microcapsules, solvent systems (ethyl acetate, n-hexane, acetone, petroleum ether and dichloromethane); surfactants (Tween-80, synperonic pe(R)/F68, cremophor RH40, sodium alginate, polyvinyl alcohol, sucrose fatty acid esters, lecithin, gelatin and arabic gum); carrier materials (glycerin monostearate, palmitic acid) were tested and identified via the PB design experiment (Table 3). The variables were as follows: X_1 - $V_{a/o}$ (mL); X_2 - $T_{\text{ultrasonic}}$ (min); X_3 - $M_{\text{synperonic pe(R)/F68}}$ (g); X_4 - $P_{\text{ultrasonic}}$ (W); X_5 - $M_{\text{glycerin monostearate}}$ (g); X_6 - T_{mixed} (min); X_7 , X_8 , X_9 , X_{10} , and X_{11} were blanks. The adequacy of the model was calculated, and the variables evidencing statistically significant effects were screened via Student's *t*-test for ANOVA (Table 4). Factors evidencing *P*-values of less than 0.05 were considered to have significant effects on the response. Therefore, they were selected for further optimization studies. The $M_{\text{glycerin monostearate}}$ (0.0111 g) was determined to be the most significant factor, followed by $M_{\text{synperonic pe(R)/F68}}$ quantity (0.0234 g) and $V_{a/o}$ (0.03 mL).

The lower probability values indicated the more significant factors on the EE. All the insignificant variables were neglected, and they kept the levels at X_2 - $T_{\text{ultrasonic}}$ (20 min), X_4 - $P_{\text{ultrasonic}}$ (380 W), X_6 - T_{mixed} (5 min). The other optimum levels of the three variables were further determined by RSM design.

Optimization by RSM Three operating parameters selected by PB design including $V_{\text{water/oil}}$ (mL), $M_{\text{synperonic pe(R)/F68}}$ (g) and $M_{\text{glycerin monostearate}}$ (g) were investigated the augmentation of encapsulation efficiency by RSM. The variables were: $V_{\text{water/oil}}$ (mL) (X_1) from 16.64 mL to 23.36 mL, $M_{\text{synperonic pe(R)/F68}}$ (g) (X_3) from 0.2 g to 1.0 g, M_{glycerin}

Table 5 The experimental design and results of response surface methodology

	X_1	X_3	X_5	EE%
1	-1(18)	-1(0.5)	-1(0.4)	21.9±0.91
2	-1	-1	1(0.6)	13.6±0.54
3	-1	1(0.7)	-1	18.6±0.82
4	-1	1	1	33.7±1.45
5	1(22)	-1	-1	27.2±1.16
6	1	-1	1	26.5±1.09
7	1	1	-1	36.7±1.38
8	1	1	1	27.1±0.93
9	-1.682(16.64)	0(0.6)	0 (0.5)	12.5±0.43
10	1.682(23.36)	0	0	21.3±0.65
11	0(20)	-1.682(0.2)	0	15.0±0.54
12	0	1.682(1.0)	0	22.8±0.72
13	0	0	-1.682 (0.2)	29.5±1.24
14	0	0	1.682 (0.8)	65.1±2.63
15	0	0	0	64.6±2.45
16	0	0	0	53.5±1.92
17	0	0	0	65.1±1.88
18	0	0	0	69.8±2.65
19	0	0	0	65.0±1.93
20	0	0	0	63.5±1.86

X_1 - $V_{a/o}$ (mL); X_3 - $M_{\text{synperonic pe(R)/F68}}$ (g); X_5 - $M_{\text{glycerin monostearate}}$ (g) (*P* < 0.05) (*n*=3)

Table 6 Analysis of variance for regression equation

Source	Sum of square	DF	Mean square	F-value	Probability (<i>p</i>)
Model	7504.6	9	833.8	12.1	0.0003
X_1	145.9	1	145.9	2.1	0.176
X_3	116.7	1	116.7	1.7	0.222
X_5	232.9	1	232.9	3.4	0.095
X_1X_3	5.3	1	5.3	0.08	0.787
X_1X_5	36.7	1	36.7	0.53	0.482
X_3X_5	26.1	1	26.1	0.38	0.551
X_1^2	3915.5	1	3915.5	57.0	<0.0001
X_3^2	3586.0	1	3586.0	52.2	<0.0001
X_5^2	473.2	1	473.2	6.9	0.025
Residual	686.9	10	68.7		
Lack of fit	540.7	5	108.1	3.7	0.089
Pure error	146.2	5	29.3		
Cor total	8191.5	19			
$R^2=0.916$	Adj $R^2=0.841$	Adeq Precision =8.893			

monostearate (g) (X_5) from 0.2 g to 0.8 g. They were generated with 3 factors and 5 levels by the principal of RSM, each of which was assessed at five coded levels (−1.682, −1, 0, +1, +1.682), as was shown in Table 5.

The results of RSM experiment were shown in Table 6 and expressed in the form of a second-order polynomials below:

$$\begin{aligned}
 EE = & -2468.43 + 174.26X_1 + 1913.21X_3 + 720.04X_5 \\
 & -4.12X_1^2 - 1577.43X_3^2 - 572.99X_5^2 - 4.07X_1X_3 \\
 & -10.71X_1X_5 + 180.63X_3X_5
 \end{aligned} \quad (6)$$

The model *F*-value was $12.14 > F_{0.01}(9, 6) = 7.98$, $P = 0.0003 < 0.001$, and the *P*-value for lack of fit was $0.0888 > 0.05$. The high *F*-value and non-significant lack of fit indicated that the model was good. The regression equation obtained from the ANOVA showed that the R^2 (multiple correlation coefficient) was 0.916 (a value > 0.75 indicates fitness of the model). It was an estimate of the fraction of overall variation in the data accounted by the model, and the model was capable of explaining 91.61% of the variation in response.

The effects of $V_{\text{water/oil}}$ (mL), $M_{\text{synperonic pe(R)/F68}}$ (g), and $M_{\text{glycerin monostearate}}$ (g) on *EE* were reported by the *P*-values

Fig. 1 Contour plot of the combined effects of $V_{a/o}$ (mL) and $M_{\text{synperonic pe(R)/F68}}$ (g) on the encapsulation efficiency. Fixed level: $M_{\text{glycerin monostearate}}$: 0.50 g

Fig. 2 Contour plot of the combined effects of $V_{a/o}$ (mL) and $M_{\text{glycerin monostearate}}$ (g) on the encapsulation efficiency. Fixed level: $M_{\text{synperonic pe(R)/F68}}$: 0.60 g

Fig. 3 Contour plot of the combined effects of $M_{\text{glycerin monostearate}}$ (g) and $M_{\text{synperonic pe(R)/F68}}$ (g) on the encapsulation efficiency. Fixed level: $V_{a/o}$: 20 mL

of the second-order polynomials (Table 6). Response surfaces for EE were shown in Figs. 1, 2 and 3 which gave the contour maps for the effect of $V_{\text{water/oil}}$ (mL), $M_{\text{synperonic pe(R)/F68}}$ (g), and $M_{\text{glycerin monostearate}}$ (g) on the EE . Response surfaces were drawn to determine the individual and interactive effect of test variable on rate of extraction. Response surface graphs were plotted between two independent variables while the remaining independent variables were kept at the zero coded level. The relationship between variables was illustrated by these response surface plots. The contour map could indicate the interaction strength in which the ellipse represented significance and the rotundity in verse (Zhong et al. 2011).

The model regression Eq. 6 indicated that three factors have positive linear effect on EE . Regression analysis of the experimental data showed that three factors had significant negative quadratic effects on EE . The combined effects of

Fig. 4 Distribution of lycopene microcapsules diameters

Fig. 5 Optical micrograph of lycopene microcapsules (1000×)

$V_{a/o}$ and $M_{\text{synperonic pe(R)/F68}}$ on the encapsulation efficiency were shown in Fig. 1. Maximum encapsulation efficiency (63.9%) was obtained at $V_{a/o}$ 20.20 mL and $M_{\text{synperonic pe(R)/F68}}$ 0.61 g. Further increase of $V_{a/o}$ led to deceleration in EE . Figure 2 indicated that maximum EE was achieved (64.3%) when $V_{a/o}$ and $M_{\text{glycerin monostearate}}$ were 20.16 mL and 0.53 g respectively, further increase these two factors led to decrease in EE of lycopene microencapsulation. The contour plot of Fig. 3 indicated that maximum EE (64.5%) occurred at $M_{\text{glycerin monostearate}}$ was 0.54 g and $M_{\text{synperonic pe(R)/F68}}$ was 0.61 g. The EE increased with $M_{\text{synperonic pe(R)/F68}}$ up to 0.61 g and further increase in $M_{\text{synperonic pe(R)/F68}}$ led to decelerate in the encapsulation efficiency. The results predicated by the model equation showed that a combination of adjusting the $V_{a/o}$ to

Fig. 6 Storage stability of lycopene microcapsules (—▲—) and lycopene (—□—) in 4°C

Fig. 7 Differential scanning calorimetry curves of lycopene microcapsules, components and physical mixture

20.14 mL, $M_{\text{synperonic pe(R)/F68}}$ to 0.61 g, and $M_{\text{glycerin monostearate}}$ to 0.54 g would favor maximum *EE*.

Verification of optimum conditions The validations of the statistical model and regression Eq. 6 were conducted by taking the $V_{a/o}$ 20.14 mL, $M_{\text{synperonic pe(R)/F68}}$ 0.61 g, $M_{\text{glycerin monostearate}}$ 0.54 g. In these optimized conditions, the predicted response for lycopene microcapsules *EE* was 64.6%, and the observed experimental value was 64.4%. These results confirmed the validity of the model and quite closed to the predicted values.

Determination of particle size The particle size distribution of the microencapsulated emulsion was measured by Mastersizer 2000 Laser Particle Size Analyzer. The average particle size of lycopene microcapsules was 182 nm (Fig. 4). Chiu et al. (2007) used γ -PGA and gelatin as the carrier materials to encapsulate, and a mean particle size of the microcapsule 38.7 μm was produced. The average particle size in our study was smaller, and this difference was probably due to the different carrier materials and the different emulsion system.

The shape of the lycopene microcapsule was observed by optical microscope. It could be found that lycopene microcapsule showed a spherical shape, smooth outer surface (Fig. 5). The results clearly showed that the core material (lycopene) to be encapsulated in spherical microcapsules with homogeneous size. The spherical microcapsules can

protect lycopene from being degraded in the presence of light and oxygen (Lu 2005).

Stability evaluation of the products With the protective of 5% mannitol (Sarbolouki and Toliat 1998; Song et al. 2007; Li and Bi 2006), freeze-dried samples of lycopene microcapsules can be retained stability under 4 °C for 2 month. Storage stability of lycopene microcapsules and lycopene in 4 °C were showed in Fig. 6. Compared with the control, the retention percentage of lycopene in microcapsules decreased slightly. The reason may be due to the effective microencapsulation of lycopene in carrier materials as mentioned above, which can effectively avoid the damage from oxygen and light et al., during storage.

The lycopene microcapsules were subjected to the thermal stability determination by DSC (Fig. 7). Compared to the curve of the physical mixture, a new peak was a transition temperature for lycopene microcapsules, and the lycopene microcapsules were found to possess high thermal stability even up to 160 °C, which may be due to the formation of a thermally stable complex between lycopene and the carrier materials (glycerin monostearate and synperonic pe(R)/F68) through electrostatic interaction.

Conclusion

This study presented a new way of preparation of lycopene microcapsules with ultrasonic emulsification. Nanometer-sized microcapsules were obtained and the microcapsules can be ensured edible safety.

RSM were employed to optimize the technological parameters for preparation of lycopene microcapsules. The final parameters of the optimized technics were as follows: $M_{\text{glycerin monostearate}}$ 0.54 g, $M_{\text{synperonic pe(R)/F68}}$ 0.61 g and $V_{a/o}$ 20.14 mL, and the *EE* had increased to 64.4%. Optical micrographs suggested that the microcapsules had a regular rounded shape and lycopene was embedded in the wall system. Mannitol was shown excellent efficacy on retaining the stability of lycopene microcapsules. Therefore, the results would be helpful to promote the application of lycopene and obtain formulations for preserving and stabilizing pharmaceuticals, nutraceuticals and food additives of high cost.

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