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Effect of maltodextrin dextrose equivalent and ultrasonic emulsification on encapsulation performance of tocochromanol from rice bran oil deodorizer distillate

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ABSTRACT: Tocopherol (Toc) and tocotrienol (T3) are two types of tocochromanol. T3 is attracting more attention to health research and is considered more beneficial than Toc. In this study, rice bran oil deodorizer distillate (RBODD) was utilized as a source of vitamin E extraction due to its high concentration of T3. Since vitamin E is water-insoluble and susceptible to oxidation, encapsulation was conducted in this investigation. The purpose of the research was to examine the effect of various dextrose equivalents (DE) of maltodextrin and ultrasonic emulsification time (UT) on tocochromanol contents and profiles in encapsulated powder. The study used tapioca starch and maltodextrin (DE-7, DE-10, and DE-16) as wall materials. Different durations of UT at 0, 30, and 60 min were investigated. The results showed that DE-10 and 0 min of UT were suitable conditions for producing the encapsulated vitamin E. The yield of encapsulation was over 90%. Moreover, encapsulation efficiency and entrapment efficiency were 89% and 44%, respectively. The obtained encapsulated vitamin E contained high tocochromanol (284 mg/g) and T3 (238 mg/g) with low moisture content (2 g/100 g) and surface oil (27 g/100 g). The encapsulated vitamin E which was extracted from RBODD exhibits bioavailability and nutritional potentials beneficial for various industries including food and pharmacy.

KEYWORDS: dextrose equivalent, encapsulation, rice bran oil deodorizer distillate, ultrasonic emulsification, tocochromanol

INTRODUCTION

Tocopherols (Toc) and tocotrienols (T3) are fat soluble molecules that belong to the group of vitamin E compound, collectively known as tocochromanol. Both Toc and T3 are composed of 4 natural isoforms: α, β, γ, and δ. Toc is characterized by a long-saturated side-chain tail, while T3, with 3 double bonds, has a shorter and more flexible tail than Toc [1]. The presence of double bonds, in conjunction with the unsaturated side chain of T3, facilitates its cellular and tissue penetration in saturated fatty layers of organs such as heart, brain, and liver [2]. In addition, T3 possesses a distinctive structure that enables its accommodation inside the lipid bilayer of the cellular membrane, hence facilitating the preservation of cellular integrity [3]. Consequently, T3 has been intensively studied during the past decade [3–5].

Natural sources of vitamin E include seeds, grains, and vegetable oils from various plants; however, high content of T3 is found in palm, annatto, and rice bran. For rice bran, it is commonly utilized as animal feed or for extracting protein [6] and a potential for oil extraction, as it contains beneficial phytochemicals that include phenolic compound, vitamin E, and γ-oryzanol [7]. In the vegetable oil industry, rice bran oil deodorizer distillate (RBODD) is a by-product of oil refinery process. RBODD is not well-known, compared with other vegetable oils, e.g., soybean oil and palm oil, primarily because of its low production [8]. In our previous study [9], it was demonstrated that the process of extracting crude vitamin E from RBODD resulted in a significantly high amount of T3 (about 80% of the tocochromanol content). Nonetheless, the utilization of fat-soluble vitamins for food supplement and fortification remains a difficulty owing to their limited solubility in water [10, 11]. The oil form exhibits limited applicability within the nutraceutical, with its usage primarily confined to formulations such as chewable tablets [12] and soft gel. For this reason, the encapsulation techniques were employed to enhance the solubility of vitamin E.

Encapsulation approach is imperative to develop delivery systems that effectively protect functional bioactive components against unfavorable processing, storage, and consumption circumstances, while also ensuring their integration within hydrophilic matrices. The encapsulation of vitamin E presents numerous advantageous features compared to its direct administration, rendering it a careful choice for ensuring maximum absorption and effectiveness [13]. Additionally, encapsulation facilitates accurate dosage regulation, so guaranteeing the administration of the desired quantity of tocochromanol while maximizing the potential for water solubility.

Among the commonly employed wall materials for encapsulation, notable options include maltodextrin, Gum Arabic, sodium caseinate, pectin, starch, chitosan, and whey protein. Maltodextrin is characterized by a dextrose equivalent (DE) value below 20. The DE value serves as an indicator of the number of glucose molecules present in maltodextrin [14]. Maltodextrins possessing a DE within the range of 5 to 20 emerge as favorable candidates for encapsulation purposes, owing to their notable attributes such as high solubility, low viscosity even at high solid concentrations, and excellent foam stability [15].

In order to enhance the encapsulation performance, it is imperative to incorporate preprocessing techniques within the encapsulation process. Among these techniques, ultrasonic emulsification is widely recognized for its ability to enhance emulsion stability by inducing acoustic cavitation, which leads to the collapse of air bubbles within the system [16]. An earlier investigation has reported that ultrasonication exhibits the potential to enhance the stability of α-Toc by offering protection against degradation induced by light and oxygen [17].

At present, there is limited study involved in extracting tocochromanol from RBODD and further producing them in a powder form. Thus, this study was conducted as a comprehensive examination of different DE of maltodextrin and ultrasonic time during emulsion preparation to produce vitamin E powder with the aim to optimize the encapsulation condition of tocochromanol. Furthermore, this study aimed to evaluate the tocochromanol profile and assess the physicochemical of the vitamin E encapsulated.

MATERIALS AND METHODS

Materials

RBODD from the chemical refinery process was considerately sponsored by Surin Bran Oil Co., Ltd., Surin province, Thailand. Food grade maltodextrin (DE-7, DE-10, and DE-16) was kindly supported by WGC Co., Ltd., Nakhon Pathom province, Thailand. Gum Arabic (Food grade) was purchased from Sigma-Aldrich Co., Ltd. (Darmstadt, Germany). HPLC-grade hexane, tetrahydrofuran, isopropanol, and dichloromethane were used, and the other chemical reagents used were of analytical grade. Tocochromanol standards: α-, β-, γ-, and δ-Toc and α-, β-, γ-, and δ-T3 were purchased from Sigma Aldrich Co., Ltd. and Eisai Food & Chemical Co., Ltd. (Tokyo, Japan).

Vitamin E extraction

Vitamin E was extracted from RBODD using ethanol by ratio 1.95 (w/v) according to our previous study [9]. The extraction mixture was refluxed at 80 ± 5 °C for 30 min before incubation at low temperature −26 °C for 24 h. Vitamin E extract (VEE) was prepared through the process of evaporating ethanol at 40 °C under vacuum condition using a rotary evaporator. VEE was stored in amber glass vials at −20 °C until analyzed.

Encapsulation of vitamin E

VEE encapsulation method was modified from Sahlan et al [18]. VEE from RBODD was utilized as a core material for encapsulation process. The experimental design was 4*3 Factorial with 4 different DE (0, 7, 10, and 16), while tapioca starch DE-0 was used as the control with 3 durations of ultrasonic emulsification time (UT: 0, 30, and 60 min). Gum Arabic and maltodextrin (DE 0, 7, 10, and 16) were mixed at a ratio of 1:1 and used as wall materials. Dissolving wall materials with distilled water at a ratio of 1:2 (w/v) and homogenizing at 5,000 rpm for 5 min produced a wall material solution. Then, VEE was added to the wall material solution at a ratio of 1:1 (w/w) and homogenized at 10,000 rpm for 5 min. Emulsification was performed by using a homogenizer (model IKA-T-25-D, IKA, North California, USA). Ultrasonic bath 150 watt at 37 kHz (Elma Elmasonic S 60 H, Elma Schmidbauer GmbH, Germany) was used for 0, 30, and 60 min at temperature of 30 ± 2 °C to assist emulsion preparation following by soaking in water bath at the same temperature for 60, 30, and 0 min, respectively. The emulsion samples were then frozen at −40 °C for 18 h and lyophilized at −80 °C for 24 h. The encapsulated vitamin E samples obtained were ground to powder by blender (model HGB2WT, Waring Commercial, Torrington, USA), sieved through a 20 mesh (850 µm), and collected in vacuum aluminium bags until analyzed. The final vitamin E encapsulated powder (VEP) samples were then obtained.

Entrapment efficiency determination

The quantification of total oil (TO) and surface oil (SO) was conducted using a modified methodology based on the approach outlined by Karrar et al [19]. In this experiment, 1.5 g of VEP was dissolved in 20 ml hexane. The mixture was gently agitated manually for 2 min at 25 °C to facilitate the extraction of SO. After filtration, the remaining substance was washed by using

30 ml hexane. The entire process was repeated twice. The mixed filtrates were evaporated at 45 °C using a rotary evaporator to remove the hexane. The content of SO was determined based on the observed weight reduction. The Soxhlet technique was employed for the extraction of TO.

The entrapment efficiency (ETE) was calculated according to the following equations:

$$\text{ETE (\%)} = \frac{\text{TO} - \text{SO}}{\text{TO}} \times 100 \quad (1)$$

where TO = total oil of vitamin E encapsulated powder (mg/100 g) and SO = surface oil or unencapsulated oil of vitamin E encapsulated powder (mg/100 g).

Moisture content

The moisture content of encapsulated vitamin E was measured gravimetrically by oven drying at 105 °C until a constant weight was achieved by AOAC method 927.05 [20].

Vitamin E (tocochromanol) determination

The quantitative analysis of tocochromanol was modified from Yuenyong et al [21]. The analysis was conducted using an Agilent HPLC 1100 with a fluorescence detector (Model 1046A, Hewlett Packard, California, USA). The separation column employed was VertiSep™ UPS silica column (4.6 × 250 mm, 5 µm, Vertical Chromatography Co., Ltd., Bangkok, Thailand). The mobile phase consisted of a mixture of hexane, tetrahydrofuran, and isopropanol (in a ratio of 93:6:1) using an isocratic elution method. The column was held at a consistent temperature of 30 °C, while the flow rate was 0.5 ml/min. The detection of tocochromanol was accomplished using fluorescence technique with excitation at a wavelength of 294 nm and emission at 326 nm. Then, 1 g/ml of the sample in dichloromethane was prepared and filtered through a 0.45 µm syringe filter. The chromatographic analysis employed a sample injection volume of 5 µl.

Encapsulation efficiency and yield determination

The Encapsulation Efficiency of Vitamin E (EEV) and yield were calculated according to the following equations:

$$\text{EEV (\%)} = \frac{\text{Tocochromanol}_{\text{VEP}}\ (\text{mg/g})}{\text{Tocochromanol}_{\text{VEE}}\ (\text{mg/g})} \times 100 \quad (2)$$

$$\text{Yield (\%)} = \frac{\text{Dried weight of VEP (g)}}{\text{Weight of emulsion in formula (g)}} \times 100 \quad (3)$$

where VEP = vitamin E encapsulated powder and VEE = vitamin E extract from RBODD.

Scanning electron microscope (SEM)

The scanning electron microscope (LEO 1455VP, Carl Zeiss, Oberkochen, Germany) was utilized to analyze the morphological and microstructural characteristics of VEP. The VEP samples were coated with a layer of gold and afterwards examined under high vacuum conditions using an accelerator voltage of 10.00 kV. The digital images were acquired at a magnification level of 1000X.

Table 1 Effect of different DE and UT on encapsulated powder yield and TO content.

Parameter		Yield (%)	Total Oil (g/100 g of powder)
DE	0	91.80 ± 0.35^{b}	48.62 ± 0.50^{b}
	7	91.91 ± 0.56^{b}	49.29 ± 0.61^{a}
	10	92.73 ± 1.16^{a}	49.08 ± 0.74^{ab}
	16	93.00 ± 0.67^{a}	48.55 ± 0.59^{b}
UT (min)	0	92.71 ± 0.82^{x}	48.86 ± 0.70^{NS}
	30	91.91 ± 0.87^{y}	48.65 ± 0.67^{NS}
	60	92.46 ± 0.84^{xy}	49.14 ± 0.59^{NS}

Value expresses as mean ± SD with 3 replicates, and the values with the different alphabets are significantly different ($p < 0.05$) using DMRT: abc for comparing DE of maltodextrin and xyz for comparing UT. No interaction was observed between DE and UT at $p < 0.05$. Therefore, these values were calculated from the main effect of DE (0, 7, 10, and 16) and UT (0, 30, and 60). NS: no significant difference, DE: dextrose equivalent of maltodextrin, and UT: ultrasonic time.

Statistical analysis

The collected data were set up in a 4 × 3 factorial design (2 factors, namely, DE and UT) and then subjected to Two-way analysis of variance. The analysis was conducted with SPSS 17. Significant differences ($p < 0.05$) between samples were evaluated using Duncan's new multiple range test (DMRT). Three replicates were performed in the experiment. PLS coefficient assessments were performed to determine the effect of DE and UT on the concentrations of tocochromanol and γ-T3. Leave-One-Out Cross-Validation (LOOCV) technique was used for building PLS regression. This analysis was carried out using custom MATLAB scripts (MATLAB V10.0, The Math Works Inc., Natick, USA). The predictive factors utilized were the relationships between DE and UT, whereas the responses measured were the concentration of tocochromanol and γ-T3. Standardization was employed for data preprocessing in order to normalize the impact of each variable on the model evaluation [22].

RESULTS AND DISCUSSION

Encapsulation yield

The encapsulation yield results indicated that there is no interaction between the DE and UT. The yield values ranged from 91.80 to 93.00% for DE and 91.91 to 92.71% for UT, as shown in Table 1. These percentages, exceeding 90%, indicated that the present encapsulation process was highly effective for producing powdered vitamin E from RBODD. In a previous study, Šturm et al [23] found that freeze drying produced a

maximum 80% yield of non-dewaxed propolis encapsulated using Gum Arabic as wall material. However, the present study used Gum Arabic and maltodextrin at a ratio of 1:1 as wall material, which provided a higher yield.

Entrapment efficiency (ETE)

TO content represented the whole amount of VEE from RBODD present within the VEP, whereas SO content referred to VEE from RBODD that unencapsulated in VEP. Ideal encapsulation requires a high TO and a low SO. The term "entrapment efficiency" refers to the effectiveness of encapsulating VEE from RBODD contained within VEP. Interestingly, there was no interaction found for TO (Table 1), while SO was affected by interaction between the DE and UT (Table 2).

TO was individually affected by either DE or UT. TO content ranged from 48.55 to 49.29 g/100 g of powder for DE and 48.65 to 49.14 g/100 g of powder for UT (Table 1). These results indicated that the TO values were nearly equal to the initial core material amount (50 g/100 g) used in the encapsulation formula. The presence of a high TO suggested that the encapsulation process was successful and effective when employing both parameters along with the freeze-drying technique.

SO content ranged from 26.49 to 39.12 g/100 g of powder (Table 2). It should be noted that the SO content reveals an interaction between DE and UT, demonstrating how the combination of these factors affects the SO content. The SO appeared to decrease with increasing DE levels. This suggests that the combined effects of higher DE and varying UT lead to reduced SO content. The lowest SO content was observed in sample numbers 8 and 9 (DE-10 with UT at 30 and 60 min, respectively). This finding agreed with the results reported by Zhu et al [24], which demonstrated lower SO values when using DE-10 compared to DE-15 and DE-20. Additionally, the previous study [25] using DE-10 showed low SO compared to DE-5. However, with increasing DE values, due to the reduction in average molecular weight of maltodextrin, the mechanical strength of the encapsulating film is substantially weakened, which consequently results in higher SO content [24]. The SO content is highest when using DE-0, followed by DE-7 and DE-16. Therefore, the average level of DE (DE-10) was recommended as an effective maltodextrin for obtaining low SO content in VEP.

This efficiency is primarily due to the decrease in the average molecular weight of maltodextrin as the DE value increases. The reduced molecular weight results in diminished mechanical strength of the encapsulating film, which consequently leads to an increased SO content following the spray-drying process.

According to Eq. (1), ETE was calculated based on TO and SO content. The ETE values indicated an interaction between DE of maltodextrin and UT, ranging from 19.00 to 45.41% (Table 2). This interaction suggests that the impact of ultrasonic time on ETE is dependent on the level of DE used. The ETE trend appeared to have increased after the DE was increased. Higher percentages of ETE reflected the superior performance of encapsulating the core material. The highest percentage was particularly evident when using DE-10 in combination with all durations (0, 30, and 60 min) of ultrasonic treatment and DE-16 UT-60. Additionally, since ETE is calculated as the percentage of the difference between TO and SO relative to TO (Eq. (1)), lower SO values observed with DE-10 and DE-16, regardless of UT conditions, suggest higher ETE values.

Moisture content

The moisture content (MC) of freeze-dried vitamin E from RBODD powder is presented in Table 2. MC ranged from 1.38 to 2.39%. These findings indicated that MC of the VEP was lower than the maximum moisture specification (3–4%) required for dried powder production in the food industry [25]. Additionally, Li et al [26] reported that microcapsules require the maximum moisture content (4–6%) of food powder that is appropriate for long-term storage. The low MC could be attributed to the ultrasound potential to break down the air bubbles in the emulsion structure, thereby making the emulsion more compact and leaving less space for water molecules to be trapped in the microstructure after freeze-drying.

Encapsulation efficiency of vitamin E

Analysis of VEE from RBODD revealed the presence of 7 derived compounds of tocochromanol as shown in Fig. S1, including 4 Toc and 3 T3, except for β-T3. It should be noted that β-T3 was not detected in the RBO sample. This finding agrees with a previous study by Pokkanta et al [27] that discovered no β-T3 in 14 different types of RBO samples. It could be also due to the low β-T3 concentration in the sample. In addition, previous research by Endo and Nakagawa [28] discovered no presence of β-T3 (0 mg/100 g) in crude RBO from Thailand.

As for the 3×4 factorial analysis in CRD for different measured parameters, it was found that most of parameters, except encapsulation yield and TO, exhibited interaction between DE and UT. Table 3 provides the concentration range found for each derived Toc and T3 compound. Notably, γ-T3 exhibited the highest concentration (115.53–224.15 mg/g) compared to the other 7 compounds with most of these compounds being present in the samples number 7 using DE-10 and no ultrasonic time (UT-0). The results in Table 3 show that the majority of measured responses increased as DE increased from DE-0 to DE-7, then results showed slight changes for DE-10. Low values of responses were

Table 2 Effect of different DE of maltodextrin and UT on surface oil, encapsulation efficiency, and moisture content of encapsulated vitamin E powder.

Treatment	Surface oil (g/100 g of powder)	Entrapment efficiency (%)	Moisture content (%)
1. DE-0 UT-0	39.12 ± 0.25^{a}	19.00 ± 0.54^{f}	2.04 ± 0.09^{b}
2. DE-0 UT-30	36.90 ± 0.29^{b}	24.66 ± 0.43^{e}	1.56 ± 0.04^{e}
3. DE-0 UT-60	36.09 ± 0.27^{c}	25.73 ± 0.75^{e}	2.04 ± 0.05^{b}
4. DE-7 UT-0	30.36 ± 0.17^{d}	38.47 ± 1.11^{d}	1.55 ± 0.04^{e}
5. DE-7 UT-30	30.39 ± 0.52^{d}	37.79 ± 1.20^{d}	1.48 ± 0.01^{ef}
6. DE-7 UT-60	30.23 ± 0.11^{d}	39.12 ± 0.13^{d}	1.68 ± 0.04^{d}
7. DE-10 UT-0	27.51 ± 0.18^{ef}	44.29 ± 0.39^{ab}	1.65 ± 0.03^{d}
8. DE-10 UT-30	26.49 ± 0.31^{g}	45.41 ± 0.78^{a}	1.42 ± 0.06^{fg}
9. DE-10 UT-60	26.94 ± 0.36^{fg}	45.38 ± 1.31^{a}	1.38 ± 0.04^{g}
10. DE-16 UT-0	27.78 ± 0.33^{e}	42.61 ± 0.35^{c}	2.39 ± 0.08^{a}
11. DE-16 UT-30	27.51 ± 0.63^{ef}	43.00 ± 0.64^{bc}	1.77 ± 0.05^{c}
12. DE-16 UT-60	27.24 ± 0.19^{ef}	44.38 ± 1.10^{ab}	2.33 ± 0.02^{a}

Value expresses as mean ± SD with 3 replicates, and the values with the different alphabets are significantly different ($p < 0.05$) using DMRT.

Table 3 Effect of different DE of maltodextrin and UT on vitamin E content and encapsulation efficiency of encapsulated vitamin E (EEV).

Treatment	Toc = α-Toc + β-Toc + γ-Toc + δ-Toc (mg/g)					T3 = α-T3 + γ-T3 + δ-T3 (mg/g)				Tocochromanol	EEV
	α-Toc	β-Toc	γ-Toc	δ-Toc	Toc	α-T3	γ-T3	δ-T3	T3	(mg/g)	(%)
1. DE-0 UT-0	10.57 ± 0.25^{e}	1.36 ± 0.04^{j}	15.07 ± 0.39^{g}	1.96 ± 0.07^{e}	28.96 ± 0.63^{h}	1.70 ± 0.01^{b}	133.14 ± 1.07^{g}	8.09 ± 0.07^{g}	142.94 ± 1.15^{g}	171.90 ± 1.71^{h}	54.24 ± 0.54^{h}
2. DE-0 UT-30	10.29 ± 0.40^{ef}	1.15 ± 0.04^{k}	12.77 ± 0.43^{j}	2.17 ± 0.09^{d}	26.38 ± 0.40^{j}	0.78 ± 0.02^{h}	115.53 ± 3.18^{i}	5.43 ± 0.15^{i}	121.74 ± 3.35^{i}	148.12 ± 3.59^{j}	46.73 ± 1.13^{j}
3. DE-0 UT-60	11.91 ± 0.43^{d}	1.97 ± 0.06^{g}	15.68 ± 0.45^{f}	1.63 ± 0.08^{g}	31.19 ± 0.74^{g}	1.20 ± 0.02^{e}	131.46 ± 2.54^{g}	5.57 ± 0.11^{i}	138.23 ± 2.67^{h}	169.42 ± 1.94^{h}	53.45 ± 0.61^{h}
4. DE-7 UT-0	9.98 ± 0.19^{efg}	1.64 ± 0.01^{i}	15.77 ± 0.12^{f}	2.05 ± 0.05^{de}	29.44 ± 0.15^{h}	0.83 ± 0.02^{g}	146.21 ± 3.07^{f}	8.36 ± 0.18^{f}	155.40 ± 3.26^{f}	184.83 ± 3.35^{g}	58.32 ± 1.06^{g}
5. DE-7 UT-30	9.77 ± 0.04^{fg}	1.82 ± 0.01^{h}	13.38 ± 0.04^{i}	1.92 ± 0.05^{ef}	26.89 ± 0.11^{ij}	0.81 ± 0.00^{gh}	126.78 ± 0.26^{h}	7.45 ± 0.02^{h}	135.04 ± 0.28^{h}	161.93 ± 0.17^{i}	51.09 ± 0.05^{i}
6. DE-7 UT-60	9.54 ± 0.13^{g}	1.76 ± 0.02^{h}	14.51 ± 0.20^{h}	1.80 ± 0.04^{f}	27.60 ± 0.13^{i}	0.94 ± 0.01^{f}	134.73 ± 1.16^{g}	7.37 ± 0.06^{g}	143.03 ± 1.24^{g}	170.64 ± 1.17^{h}	53.84 ± 0.37^{h}
7. DE-10 UT-0	16.74 ± 0.25^{a}	2.82 ± 0.06^{a}	24.29 ± 0.55^{a}	2.76 ± 0.12^{a}	46.60 ± 0.63^{a}	1.50 ± 0.01^{c}	224.15 ± 2.13^{a}	12.36 ± 0.12^{a}	238.01 ± 2.26^{a}	284.61 ± 2.40^{a}	89.80 ± 0.76^{a}
8. DE-10 UT-30	16.34 ± 0.51^{a}	2.50 ± 0.03^{b}	21.01 ± 0.25^{c}	2.59 ± 0.08^{b}	42.44 ± 0.76^{c}	1.33 ± 0.02^{d}	202.14 ± 2.38^{c}	10.91 ± 0.13^{c}	214.37 ± 2.52^{b}	256.81 ± 3.27^{b}	81.03 ± 1.03^{c}
9. DE-10 UT-60	16.21 ± 0.53^{a}	2.75 ± 0.03^{c}	21.80 ± 0.25^{b}	2.80 ± 0.13^{a}	43.56 ± 0.87^{b}	2.04 ± 0.04^{a}	208.06 ± 3.88^{b}	10.59 ± 0.20^{b}	220.69 ± 4.12^{c}	264.26 ± 4.93^{c}	83.38 ± 1.55^{b}
10. DE-16 UT-0	13.63 ± 0.20^{b}	2.40 ± 0.04^{d}	17.55 ± 0.28^{d}	2.45 ± 0.04^{bc}	36.03 ± 0.35^{d}	1.49 ± 0.01^{c}	166.99 ± 0.88^{d}	9.45 ± 0.05^{d}	177.93 ± 0.94^{d}	213.96 ± 0.86^{d}	67.51 ± 0.27^{d}
11. DE-16 UT-30	13.28 ± 0.33^{bc}	2.13 ± 0.01^{f}	17.07 ± 0.11^{d}	2.41 ± 0.07^{c}	34.89 ± 0.29^{e}	1.18 ± 0.01^{e}	146.79 ± 0.81^{f}	8.98 ± 0.05^{e}	156.95 ± 0.86^{f}	191.84 ± 0.98^{f}	60.53 ± 0.31^{f}
12. DE-16 UT-60	12.85 ± 0.43^{c}	2.27 ± 0.05^{e}	16.40 ± 0.38^{e}	2.36 ± 0.12^{c}	33.88 ± 0.92^{f}	1.47 ± 0.03^{c}	156.55 ± 2.74^{e}	8.91 ± 0.16^{f}	166.93 ± 2.92^{e}	200.82 ± 3.83^{e}	63.36 ± 1.21^{e}

Value expresses as mean ± SD with 3 replicates, and the values with the different alphabets are significantly different ($p < 0.05$) using DMRT. DE: dextrose equivalent of maltodextrin, UT: ultrasonic emulsification time, Toc: tocopherol, T3: tocotrienol, and Tocochromanol = Toc + T3.

observed when using DE-16.

From Table 3, the utilization of ultrasound-assisted encapsulation has demonstrated that the absence of ultrasound (0 min duration) provided higher concentrations of tocochromanol derivatives in comparison to durations of 30 and 60 min. In general, ultrasound enhances the stability of the emulsion and its microstructure by inducing acoustic cavitation, resulting in the collapse of air bubbles contained within the emulsion. This process contributes to the improvement of the stability of the encapsulated powder [16]. Nonetheless, our findings indicated that interaction between DE and UT leads to a reduction in the concentration of tocochromanol derivatives within the encapsulated product. This observation could be attributed to a loss of concentration occurring during the ultrasound emulsification procedure. It should be noted that the significant shear forces generated by cavitation bubbles during ultrasound emulsification have the potential to cause negative effects on delicate structures or sensitive ingredients present within the emulsion, including vitamin E [29].

Tocochromanol content was calculated as a grand total of Toc and T3 in the encapsulated vitamin E and ranged between 148.12–284.61 mg/g. The tocochromanol present in the encapsulated vitamin E was utilized to determine the EEV according to Eq. (2), which ranged from 46.73 to 89.80% (Table 3). The contribution of each derived tocochromanol compounds, Toc, T3, tocochromanol, and EEV, was specifically observed to be a high amount in treatment using DE-10. Furthermore, DE-10 exhibited higher EEV compared to other DE of maltodextrin.

There is an inverse relationship between the average molecular weight of maltodextrin and its DE value. Lower-molecular weight maltodextrins have high DE and are composed of shorter chains. The incorporation of high-DE maltodextrin resulted in a substantial decrease in the elasticity of the film, hence leaving it unsuitable for the purpose of encapsulating vegetable

oil [23]. However, low DE of maltodextrin may have limited matrix stability, meaning that the encapsulated materials may be more prone to physical and chemical degradation. The lower degree of hydrolysis in low DE of maltodextrin results in fewer available hydroxyl groups for cross-linking or interactions with the encapsulated materials. Consequently, this could lead to decreased protection and stability of the encapsulated materials during storage or processing [30]. Thus, DE-10, being positioned between the low and high DE ranges of maltodextrin, was considered the most efficient option for encapsulation of VEE in this study. The significance of using maltodextrin with a particular DE as a wall material in the encapsulation process depends on its functional properties, resulting in maltodextrin being an ideal material for encapsulating a wide range of substances. Higher DE values result in a more water-soluble maltodextrin, which accelerates the release of the encapsulated substance when exposed to water. Alternatively, lower DE values (DE-0) produce less water-soluble maltodextrin, resulting in a delayed and more controlled release of the encapsulated substance [31].

This study discovered that VEP contains 7 vitamin E compounds, including α-, β-, γ-, and δ-Toc and α-, γ-, and δ-T3, the new food or pharmaceutical ingredient. In the majority of previous investigations, α-Toc was used as the core material for encapsulating vitamin E [11, 32–34]. In addition, γ-T3 had a very high concentration in this study compared to other compounds with up to 75% in tocochromanol or vitamin E. Phang et al [5] reported that γ-T3 may be preferable to Toc in terms of biological activities due in part to structural differences. T3 unsaturated bonds and shorter side chains enable increased fluidity and uniform distribution within the phospholipid bilayer. In addition, α-Toc is retained preferentially by body tissues via α-Toc transfer protein, whereas γ-T3 are swiftly degraded to short-chain carboxy chromanols and conjugated counterparts, which have been demonstrated to have superior biological effects. This suggests that γ-T3 are more effective at scavenging peroxyl radicals than α-Toc due to a more effective interaction in membrane environments.

Chemometric studies of the effect of DE and UT on tocochromanol and γ-T3 concentrations

The effect of DE and UT on the concentrations of tocochromanol and γ-T3 in encapsulated vitamin E from RBODD is illustrated (Fig. 1). The results of the investigation revealed that both tocochromanol and γ-T3 exhibited the maximum concentration in samples, ranging from 250–300 mg/g for tocochromanol and 200–250 mg/g for γ-T3, when utilizing DE-10 with all UT. The difference in concentration between samples utilizing distinct DEs was readily apparent, signifying that the selection of DE had a substantial influence on the concentration of both substances. On the contrary,

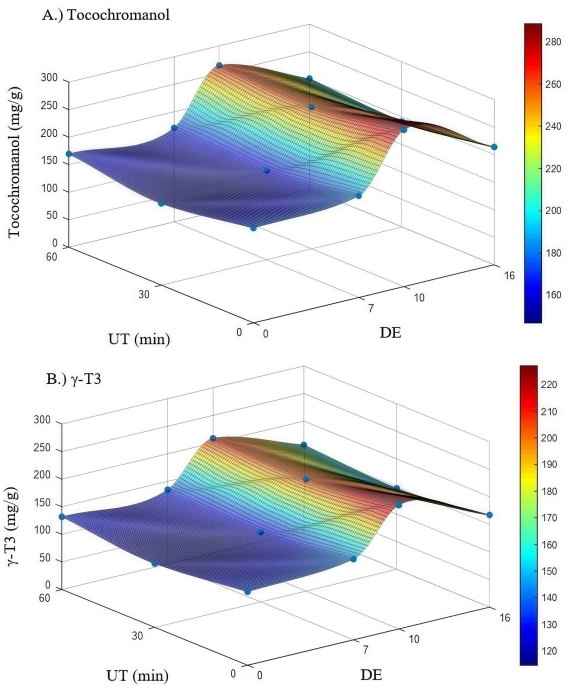

Fig. 1 3D surface plot of difference between DE and UT on (a) tocochromanol and (b) γ-T3.

the utilization of UT had a negligible effect on them. In order to develop a comprehensive understanding of the effects of both DE and UT, a chemometric technique utilizing Partial Least Squares (PLS) needed to be employed.

A PLS model was developed to analyze the content of tocochromanol and γ-T3 in encapsulated vitamin E, using the difference between DE and UT. The prediction results for tocochromanol showed that the low root mean square error of cross validation (RMSECV) value confirmed the model accuracy and suitability for prediction purposes. Furthermore, it was anticipated that the root mean square error of calibration (RMSEC) would be less than the RMSECV, as the errors obtained from the missing samples are expected to be higher than when the model was used to forecast itself in the auto prediction test [34]. This suggests that the model was not susceptible to the issue of over-fitting. In addition, the low R^2 and Q^2 values indicate that the model does not match the data well, implying that there may be additional factors affecting the accuracy of the predictions. Nevertheless, the low RMSECV value instilled confidence in the model capacity to precisely forecast the target variable. This suggests that the model is resilient and possesses a strong ability to apply its knowledge to many situations, making it well-suited for actual use. In general, the findings confirmed the efficacy of the model in predicting the intended result.

The PLS coefficients indicated the significance of the parameters in predicting the response. The values of the coefficients can be utilized to determine the degree of significance or influence that the variables have on the prediction models. The PLS coefficients of the DE parameter in both the tocochromanol and γ-T3 models, as shown in Fig. S2, indicated a positive and significant influence on concentration. Additionally, there is a slight positive effect observed from the interaction between DE and UT (DE*UT). Increases in the quantity of DE led to a corresponding increase in the concentration of tocochromanol and γ-T3. In comparison to DE, the coefficient of UT had a negative value and was rather tiny, suggesting that the use of UT had a reverse impact on the levels of both compounds. Furthermore, the interaction term DE*UT indicates that the impact of DE on compound concentration is altered by the presence of UT. More precisely, when the quantity of DE increases, the impact of UT becomes less noticeable. Consequently, it may be inferred that DE has an advantageous effect on compound concentration, whereas UT has a declining influence on it.

Scanning electron microscope (SEM)

Fig. S3 shows SEM images illustrating the encapsulated vitamin E from RBODD using different DE of maltodextrin and UT. These images brightly demonstrated the formation of porous structures in the encapsulated vitamin E. Throughout the freezing phase, the microstructure remains largely unchanged, except for the emergence of pores resulting from water removal within the particles. It was a common occurrence for microcapsules to exhibit numerous surface pores due to the sublimation of water during the freeze-drying process [35].

Additionally, the pores size appeared to be influenced by the specific DE values of the maltodextrins utilized, leading to a more uniform surface coverage and compact structure. In the present study, DE-7, DE-10, and DE-16 exhibited particles with highly porous structures. The relatively limited number of pores observed in the structure can be attributed to the pores being filled with flaxseed oil that freezes on the particle surface, as highlighted in a prior investigation conducted by Elik et al [36]. This clarification corresponded with the current study as it utilized VEE, having the same oil form as flaxseed oil.

CONCLUSION

The study examined the impact of different DE of maltodextrin and UT on the encapsulation of vitamin E from RBODD. Analysis of the vitamin E compounds revealed the presence of 7 derived compounds, with γ-T3 exhibiting the highest concentration. γ-T3 contributed up to 75% of tocochromanol concentration of VEP. By employing multivariate analysis, the utilization of PLS coefficients facilitated the elucidation of the properties of tocochromanol and γ-T3 by identifying the distinguishing features of DE and UT. DE-10 of maltodextrin was found to be the most efficient wall material for encapsulation, a balance between low and high DE maltodextrins, as achieving high EEV and producing high-quality vitamin E powder from RBODD. Interestingly, the duration of ultrasonic emulsification did not significantly affect the microcapsule properties. Future research should focus on investigating additional aspects of the powder quality, including particle size, chemical composition analysis, release of vitamin E, and thermal analysis.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at https://dx.doi.org/10.2306/scienceasia1513-1874.2024.101.

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Appendix A. Supplementary data

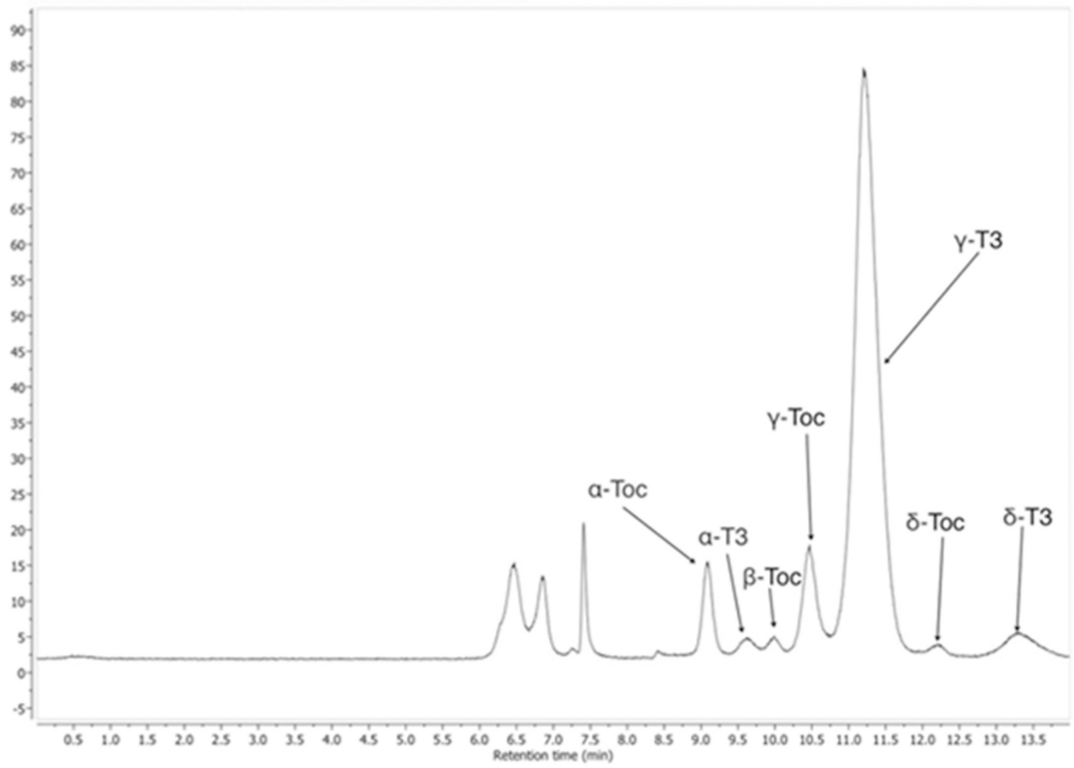

Fig. S1 HPLC chromatograms of vitamin E derivatives in vitamin E extract from RBODD.

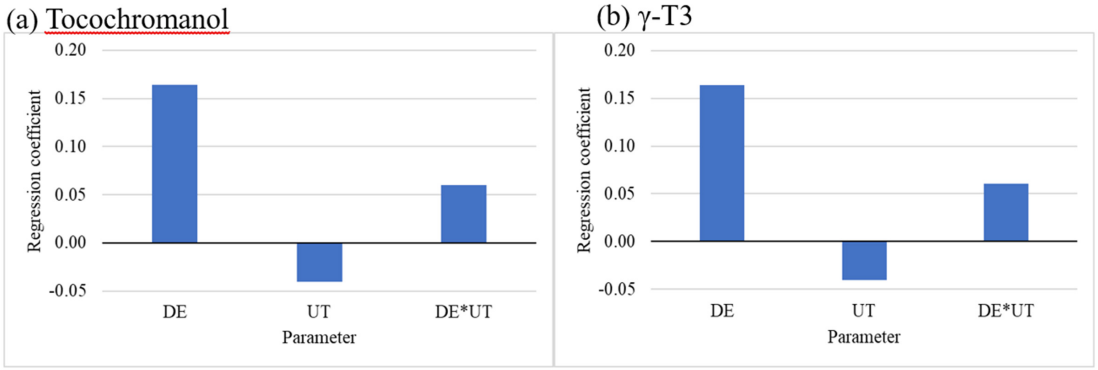

Fig. S2 PLS coefficients of the models established for each of (a) tocochromanol and (b) γ-T3.

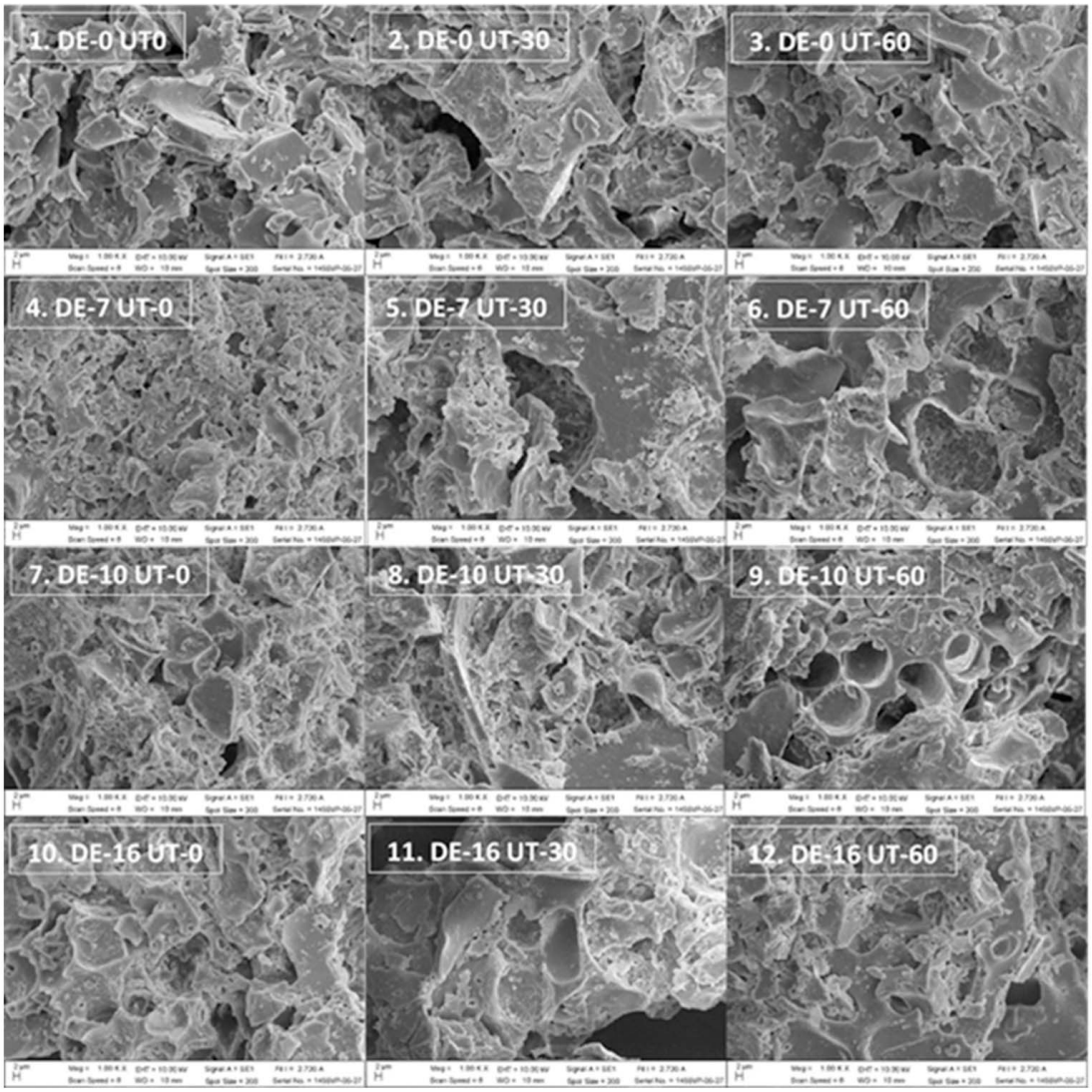

Fig. S3 SEM images of encapsulated vitamin E powder at 1000X.