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Micronization Increases Vitamin E Carrying and Releasing Abilities of Insoluble Fiber

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This study was to investigate the effects of micronization on vitamin-carrying capacity and slow-release ability of carambola (starfruit) insoluble fiber (IF) and cellulose using in vitro and in vivomodels. Upon micronization, carambola IF (8.1 μm) underwent structural changes to expose more functional groups in the fiber matrix and to exhibit higher oil-holding capacity (~20.4-fold). Micronized fibers in forms of fiber−vitamin composites, particularly the micronized carambola IF−vitamin composite, were capable of carrying vitamin E (α-tocopherol) up to 9.6-fold over their unmicronized forms and releasing nutrient gradually. Animal studies demonstrated that the adminstration of micronized carambola IF−vitamin composite could maintain the plasma vitamin E of rats at relatively higher levels (2.1−3.6-fold of the initial values) for at least 5 h. The results suggested that micronized fibers, particularly the micronized carambola IF, could be exploited as potential nutrient carriers in food applications and also be used to produce slow-release formulations.

KEYWORDS: Insoluble fiber; micronization; carambola; vitamin; carrying capacity

INTRODUCTION

Some previous studies showed that the absorption efficiency of vitamins (i.e., vitamin E) was influenced by their concentrations in the diet; for instance, the total uptake of vitamin with a single high dose was relatively lower than that with multiple low doses (*1, 2*). Total uptake of a particular nutrient is hence increased by its slow-release effect along the digestive tract. Some findings in an atherosclerosis prevention study (*3*) demonstrated that the consumption of a slow-release formulation of vitamin resulted in a beneficial effect on atherosclerotic progression. Some dietary carbohydrates also exhibited slow-release activity and led to a significant reduction in blood glucose and a flattened plasma insulin level (*4*).

Our previous studies demonstrated that an insoluble fiber material derived from the pomace of *Averrhoa carambola* (also known as carambola and starfruit) had pronounced physicochemical properties and physiological effects such as hypocholesterolemic and intestinal health-enhancement activities (*5–8*). These properties and functions could be significantly improved by particle size reduction to micrometer scale (*9, 10*). Upon micronization treatment, our recent findings have revealed that the carambola insoluble fiber particulate could be used as a potential nutrient carrier, and a novel composite prepared by the micronized fiber and vitamin E exhibited vitamin slow-release ability. It was hence interesting to investigate the nutrient-carrying and slow-release features of different fiber−vitamin composites using micronzation treatment.

In the present study, the effects of micronization treatment on the vitamin-carrying capacity and slow-release ability of different insoluble fiber materials such as carambola insoluble fiber (IF) and cellulose were investigated using both in vitro and in vivo models. Some novel fiber−vitamin composites were prepared from these insoluble fibers (unmicronized or micronized) and vitamin E. Potential applications of the fiber−vitamin composites with respect to food processing and nutritional enhancement are discussed. This study also provides insights for exploiting the potential applications of micrometer technology in the food industry.

MATERIALS AND METHODS

Preparation of Carambola Insoluble Fiber (IF). After juice extraction, carambola pomace sample was collected from CHIA-MEEI (Taiwan) Food Industrial Corp. The pomace was dried in an air oven at 40 °C for 48 h and ground to a fine powder (0.5 mm in size). The moisture content of the dried sample was 21.9 ± 0.7 g kg^{-1}. According to the method of Chau et al. (*6*), carambola IF was prepared from the powdered sample using cold distilled water as a solvent.

Micronization Treatments. In this work, plant cellulose manufactured from northeastern U.S. hardwoods such as beech, birch, and maple (Alphacel 900453, ICN Nutritional Biochemicals, Cleveland, OH) and carambola IF were used to prepare the micronized fiber samples. According to the methods of Chau et al. (*9*), these insoluble fiber samples were pulverized by a single passage of insoluble fiber samples through the milling chamber of a jet-mill (JM-1, Yenchen, Taipei, Taiwan) using compressed air at ~65 psi. After that, the fiber particulates were mixed with distilled water (1:50, w/v) and micronized

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with a high-pressure microsizer (Panda 1000, GEA, Parma, Italy) at a pressure of 11600 psi for 10 cycles. The micronized fiber slurry was then freeze-dried and kept in a desiccator until used.

Chemical Analyses and Physicochemical Properties. Moisture (method 934.01) was determined according to the AOAC method (*11*). Particle size was estimated by a laser particle size analyzer (Analysette 22-Economy, Fritsch, Germany). Oil-holding capacities (g g^{-1}) of different insoluble fiber samples were determined according to the method of Chau and Huang (*12*). The density of the vegetable oil being used was 0.85 g mL^{-1}.

Preparation of Insoluble Fiber−Vitamin E Composites. One gram of unmicronized or micronized insoluble fiber sample and 50 μg of (±)-α-tocopherol (T3251, Sigma Chemical Co., Deisenhofen, Germany) were homogenized with 200 mL of water by a single passage of the mixtures through the high-pressure microsizer (at ∼11600 psi), followed by centrifugation at 3000*g* for 20 min and freeze-dried to collect different fiber−vitamin composites including unmicronized carambola IF−vitamin (IV), unmicronized cellulose−vitamin (CV), micronized carambola IF−vitamin (MIV), and micronized cellulose−vitamin (MCV) composites. After a single passage through the homogenizer, the IV and CV composites containing unmicronized insoluble fibers were regarded as "unmicronized" samples, whereas the MIV and MCV composites containing micronized insoluble fibers were regarded as "micronized" samples.

Quantification of Vitamin E. According to the method described by Weinmann et al. (*13*), vitamin E (α-tocopherol) concentration was quantified by a HPLC system equipped with an intelligent pump (Hitachi L-2130, Tokyo, Japan) and a diode array detector (Hitachi L-2455, Tokyo, Japan) using the following conditions: a reversed-phase C-18 prepacked column (Mightysil RP-18GP, 250 mm × 4.6 mm i.d., 5 μm; Kanto Chemical, Tokyo, Japan); room temperature (25 °C); water/methanol solution (5:95, v/v) as eluent; a flow rate of 2 mL min^{-1}; and monitoring at 290 nm. The retention time was about 18.8 min, and the minimum detectable concentration was 2.5 μmol L^{-1}.

In Vitro Determination of Vitamin E Release Rate. According to the method of Shi and Tan (*14*) with some modifications, 1 g of fiber−vitamin composite was added to 100 mL of an artificial gastric juice (*15*), which was used as a dissolution medium. An aliquot (1 mL) of the mixture solution was sampled at 10 min intervals for 2 h under continuous agitation at 37 °C. After filtration, the amount of vitamin E in the filtered mixture was directly quantified by the HPLC method described above using the artificial gastric juice as a blank solution. The release rate of vitamin E from the composite was expressed by the amount of vitamin E in the filtrate as a function of time.

In Vivo Determination of Vitamin E Release Rate. The study protocol was approved by the Animal Care and Use Committee of National Chung Hsing University. Twenty-four male Sprague−Dawley rats weighing 250 ± 18 g were obtained from the National Laboratory Animal Center of Taiwan. Animals were housed (in pairs) in a stainless cage in a room maintained at 24 ± 1 °C with 12 h light/dark cycles. They had free access to regular chow diet and water. After an acclimation period of 7 days, animals were divided into six weight groups of four each. The four animals in each weight group were randomly assigned to one of the four diet groups including control-I, IV-composite, control-II, and MIV-composite groups. Control-I and -II were the corresponding controls for the IV- and MIV-composite groups, respectively. Animals in the IV- and MIV-composite groups were fed by gavage with 1 mL of corn oil in which 0.20 g of IV- and MIV-composites was added, respectively (providing 0.224 and 2.14 mg of vitamin E, respectively). Animals in the control-I and control-II groups were gavage fed with 1 mL of vitamin E-supplemented corn oil (containing 0.224 and 2.14 mg of vitamin E, respectively).

After feeding different samples by gavage, venous blood sample was drawn from the tail vein every hour for 4 h. The plasma vitamin E level was analyzed using the method of Catignani and Bieri (*16*) with slight modifications. Blood plasma (100 μL) and 50 μL of Sigma T3376 α-tocopherol acetate (52.5 mg per liter of ethanol) as an internal standard were vortex mixed for 1 min. Vitamin E in the mixture was extracted by adding 200 μL of HPLC grade hexane, followed by vortex mixing for 1 min. After centrifugation at 670*g* for 2 min, the hexane layer was collected and evaporated under nitrogen. The residue was redissolved in 50 μL of HPLC grade methanol in which vitamin E content was analyzed by the HPLC method described above. The in vivo release rate of vitamin E from the composite was estimated by the changes in plasma vitamin E concentration over time.

Table 1. Effects of Micronization on the Physicochemical Properties of Various Insoluble Fiber Materials[a]

insoluble fiber material	av particle size (μm)	oil-holding capacity (g g^{-1})
cellulose without micronization	56.2 ± 1.3w	2.72 ± 0.1w
micronized cellulose	20.9 ± 0.2x	4.85 ± 0.1x
carambola IF without micronization	266 ± 1.4y	2.95 ± 0.0w
micronized carambola IF	8.1 ± 0.1z	60.3 ± 1.3y

[a] Values (mean ± standard deviation, $n = 4$) in the same column with different letters are significantly different ($P < 0.05$).

Estimation of Vitamin E Carrying Capacity. Following the method described under In Vitro Determination of Vitamin E Release Rate with slight modifications, the vitamin E carrying capacities (mg g^{-1}) of different fiber−vitamin composites were estimated by measuring the total amount of vitamin E released into the filtrate after continuous agitation at 37 °C for 6 h.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis. According to the method described by Liu and Bai (*17*), insoluble fiber materials (approximately 1 mg) were mixed with 100 mg of potassium bromide (catalog no. 0011-184, SpectroGrade, Garfield, NJ) and pressed to form pellets at a pressure of 167 MPa. Each pellet was oven-dried at 40 °C for 24 h and then kept in a desiccator until analysis. FTIR analysis was conducted with a Bruker Equinox55 spectrometer (Billerica, MA) within the wavenumber range of ∼500–4000 cm^{-1}.

Statistical Analysis. All results were expressed as means ± standard deviation ($n = 4$) except for those from in vivo experiments ($n = 6$). All tests were analyzed by one-way analysis of variance using the Statistical Analysis System (SAS). Differences were considered to be significant at $P < 0.05$.

RESULTS AND DISCUSSION

Particle Sizes and Physicochemical Properties. Table 1 shows the effects of micronization on the average particle sizes of carambola IF and cellulose. After the pulverization by jet-milling, the average particle sizes of carambola IF and cellulose (initially at 266 and 56.2 μm, respectively) were reduced to 25.2 and 28.5 μm, respectively. The subsequent micronization treatment using a high-pressure microsizer significantly ($P < 0.05$) reduced the average particle sizes of carambola IF and cellulose to 8.1 and 20.9 μm, respectively (by ∼97.0 and ∼62.8% of their initial values). These results confirmed that the micronization treatment effectively reduced the particle sizes of insoluble fiber materials (i.e., carambola IF and cellulose) to different microsizes. As compared to the initial oil-holding capacities (OHCs) of unmicronized insoluble fiber materials, the micronization process significantly ($P < 0.05$) increased the OHCs of carambola IF and cellulose by ∼20.4- and ∼1.8-fold, respectively (**Table 1**). Proper micronization treatment effectively increased the porosity, surface area, capillary attraction, and oil-binding sites of insoluble fiber and, hence, enhanced the physical entrapment of oil inside sponge-like fiber matrix (*9*). The remarkable increase in oil retention ability of micronized carambola IF suggested its potential in being a lipid-soluble nutrient carrier and in different food applications requiring oil retention.

Structural Changes in Insoluble Fiber Materials upon Micronization. Figures 1 and **2** illustrate the effects of micronization treatment on the functional groups of different insoluble fibers by means of their FTIR spectra. As compared with the FTIR spectra of unmicronized carambola IF (**Figure**

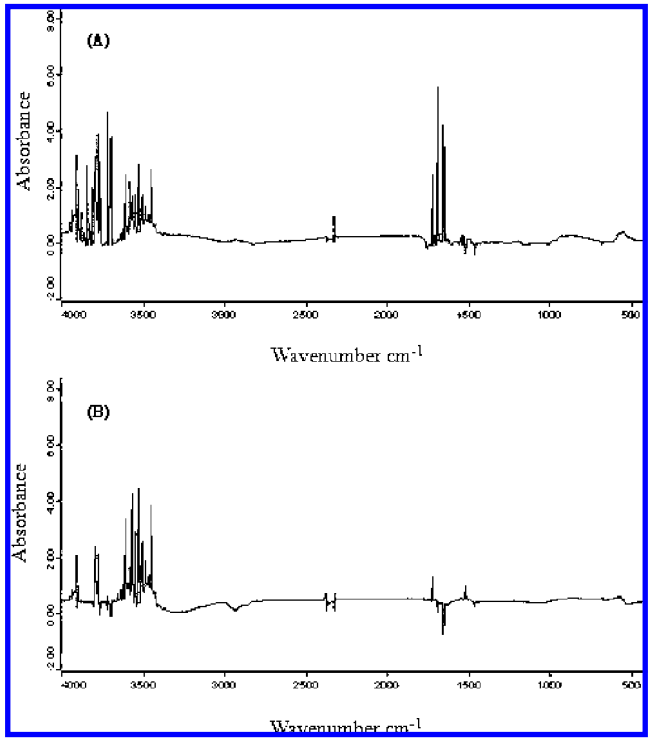

Figure 1. FTIR spectra of (**A**) micronized carambola IF and (**B**) carambola IF without micronization.

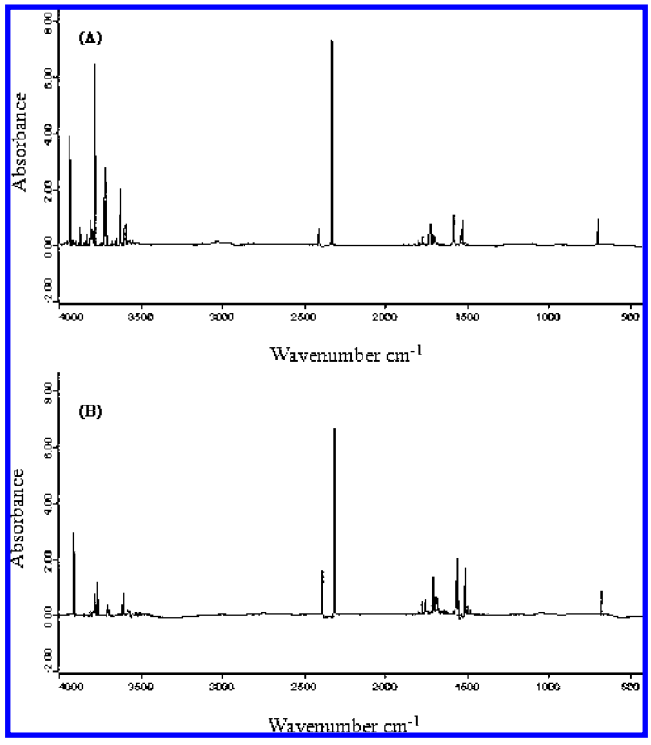

Figure 2. FTIR spectra of (**A**) micronized cellulose and (**B**) cellulose without micronization.

1), some new absorbance peaks corresponding to C═O stretch at 1655, 1690, and 1735 cm^{-1} and O—H stretch at 3500−3900 cm^{-1} were observed in the FTIR spectra of micronized carambola IF. In **Figure 2**, no apparent changes within the wavenumber range of ∼500−4000 cm^{-1} were noticed for cellulose before and after micronization. However, the results suggested that the process of micronization has exposed more functional groups such as carbonyl, carboxyl, and hydroxyl groups in the fiber matrix (*18*). The nature and position of functional groups play an important role in a wide variety of polymerization activities (*19*) as well as the ability to adsorb vitamins (*20*). The results implied that the micronization treatment could be applicable to modify the functional groups and physicochemical properties of carambola IF for different food applications.

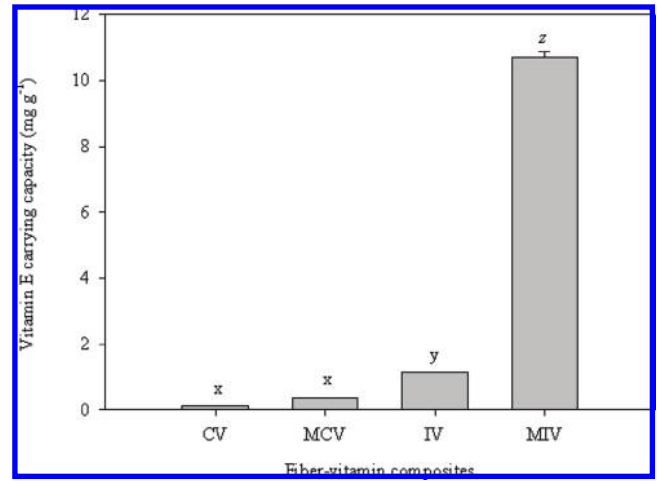

Figure 3. Vitamin E carrying capacity (mg g^{-1}) of various fiber−vitamin composites. Bars with different letters are significantly different ($P < 0.05$).

Vitamin E Carrying Capacity of Fiber−Vitamin Composites. Chemical analyses revealed that both the carambola IF and cellulose samples contained no detectable level of vitamin E. **Figure 3** presents the vitamin E carrying capacity (mg g^{-1}) of different fiber−vitamin composites. It was interesting that the insoluble fibers (with or without micronization) in forms of insoluble fiber−vitamin E composites were capable of carrying vitamin E at different extents. Without micronization, chromatographic analyses demonstrated that the levels of vitamin E carried in the IV- and CV-composites were 1.12 and 0.12 mg g^{-1}, respectively. For the composites prepared with micronized fibers, the levels of vitamin E carried in the MIV- and MCV-composites were significantly ($P < 0.05$) increased to 10.7 and 0.35 mg g^{-1}, respectively (955 and 292%, respectively). These results showed that micronization treatment could effectively increase the vitamin E carrying capacity of different fiber−vitamin composites, especially for the MIV-composite in which vitamin E carrying capacity was tremendously ($P < 0.05$) increased up to ∼9.6-fold. The ability of dietary fibers to bind vitamin E has been reported by Nnanna and O'Neill (*21*). An elevated level of vitamin E being carried in the micronized fiber−vitamin composites might be due to the highly increased oil-holding capacities of the micronized carambola IF and cellulose (**Table 1**), hence resulting in more lipid-soluble substances (i.e., vitamin E) being carried in the micronized fiber matrix. It was also inferred that the higher vitamin E carrying capacity within the micronized carambola IF was attributed to more functional groups (i.e., carbonyl, carboxyl, and hydroxyl groups) being exposed on the extended fiber surface as a result of micronization treatment. In recent years, micro- and nanoparticles have played an important role in the development of controlled release systems of bioactive ingredients. They may also be useful for the delivery of active compounds into the human body (*22*). The remarkable ability to carry vitamin E suggested that the micronized carambola IF in the form of a novel fiber−vitamin composite could be used as a promising carrier of lipid-soluble bioactive ingredient.

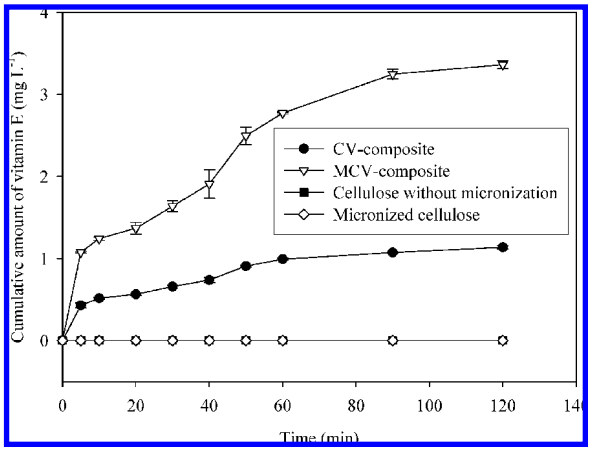

Figure 4. In vitro release rate of vitamin E from various cellulose−vitamin composites.

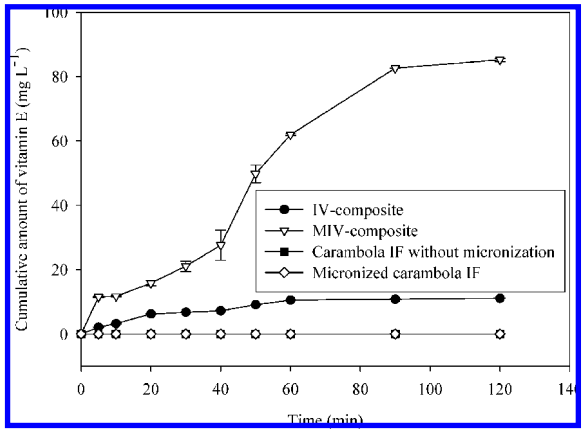

Figure 5. In vitro release rate of vitamin E from various carambola IF−vitamin composites.

In Vitro Vitamin E Releasing Activity. In **Figures 4** and **5**, the release rates of vitamin E from different insoluble fiber−vitamin composites into the artificial gastric juice are shown as a function of time (0−2 h). For the insoluble fiber materials alone, the absence of vitamin E in the insoluble fiber materials might explain the unchanged vitamin E content over time. For different fiber−vitamin composites, the increasing cumulative contents of vitamin E over every 10 min interval indicated that vitamin E was continuously released from the composites (e.g., IV-, MIV-, CV-, and MCV-composites) at different extents. Over the 2 h period, the cumulative contents of vitamin E released from these composites were gradually increased over time (**Figures 4** and **5**). Moreover, the cumulative contents of vitamin E released from the MIV- and MCV-composites were markedly higher than those from the IV- and CV-composites. This might be due to the fact that the initial levels of vitamin E being carried in the MIV- and MCV-composites were much higher (~9.6- and 2.9-fold, respectively) than those in the IV- and CV-composites (**Figure 3**). At 2 h, the cumulative amounts of vitamin E released into the gastric juice from the MIV- and MCV-composites (85.2 and 3.36 mg L^{-1}, respectively) were about 3.0−7.7-fold higher than those from the IV- and CV-composites (11.1 and 1.14 mg L^{-1}, respectively) (**Figures 4** and **5**). These results demonstrated that the micronized fiber−vitamin composites were capable of releasing more vitamin E gradually and continuously over the 2 h period. This in vitro study demonstrated that micronized insoluble fibers, especially the micronized carambola IF, in the form of fiber−vitamin composites had a remarkable ability to entrap lipid-soluble substances as well as to release the substances gradually, implying that the MIV-composite could be used as a potential carrier of bioactive ingredients and also in slow-release formulations for certain food applications.

Table 2. In Vivo Changes of Plasma Vitamin E Levels (Micromoles per Liter)[a] in Rats after the Administration of Different Carambola Fiber−Vitamin Composites

	treatments			
	unmicronized		micronized	
time(h)	control-I[b]	IV-composite[b]	control-II[c]	MIV-composite[c]
0	27.0 ± 2.7v	28.2 ± 1.5v	26.4 ± 2.5v	24.7 ± 2.4v
1	52.6 ± 3.9w	44.9 ± 4.3w	100 ± 6.7w	51.7 ± 3.2w
2	30.0 ± 3.4v	39.2 ± 1.7x	37.8 ± 3.6x	76.9 ± 5.2x
3	27.1 ± 2.2v	29.7 ± 2.3v	28.0 ± 1.9v	88.7 ± 6.4y
4	27.0 ± 1.9v	27.2 ± 0.9v	26.7 ± 2.0v	55.4 ± 5.5w
5	27.2 ± 1.1v	27.4 ± 0.6v	26.4 ± 1.6v	37.7 ± 3.6 z

[a] Values (mean ± standard deviation, $n = 6$) in the same column with different letters are significantly different ($P < 0.05$). [b] The total vitamin E intakes for the IV-composite group and its corresponding control (control-I) are 0.224 mg. [c] The total vitamin E intakes for the control-II and MIV-composite groups are 2.14 mg.

From our previous findings (*5, 9*), the micronized carambola IF, which had a highly extended, porous, and sponge-like fiber surface, possessed a high level of anionic rhamnose-rich pectic polysaccharides (above 400 g kg^{-1} of fiber). The ability of the anionic carambola fiber matrix to release vitamin as a function of time was in agreement with the findings from Ivan et al. (*23*) that an anionic polysaccharide material containing glucuronic acid in a polymer chain was used to produce slow-release formulations. Supplying a large single dose of vitamin is not always an option because of the reduction in absorption efficiency (*1, 2*); the slow-releasing characteristics of the micronized fiber composites, especially the MIV-composite, might then provide a strategy to improve the absorption efficiency of vitamin E by releasing the nutrient over a longer time in small quantities.

In Vivo Vitamin E Releasing Activity. On the basis of the potential vitamin-carrying and slow-release abilities of carambola IF upon micronization, the effects of different fiber−vitamin composites (i.e., IV- and MIV-composites) on the plasma vitamin E concentrations in rats were evaluated over a period of 4 h (**Table 2**). At 0 h, the plasma vitamin E levels of rats among different diet groups were found to be within the normal range of plasma vitamin E levels (16−33 μmol L^{-1}) (*24, 25*). Chromatographic analyses revealed that the plasma vitamin E levels of rats in the control-I and control-II groups rose to their peak values (~2.0- and 3.8-fold of the initial levels, respectively) in the first hour and then returned to normal in 2 h. For the IV-composite group, the plasma vitamin E levels reach the peak level (1.6-fold of the initial level) in the first hour and descended to the original concentration in the third hour. Over the experimental period (1, 2, 3, and 4 h), the MIV-composite could effectively ($P < 0.05$) maintain a 2.1-, 3.1-, 3.6-, and 2.2-fold increase of plasma vitamin E levels, respectively, while reaching an optimum level of plasma vitamin E in the third hour. As compared with the control-II, the slow-release characteristics of the MIV-composite help increase the plasma vitamin E at relatively higher levels for better utilization for at least 5 h. These in vivo results were in agreement with our in vitro findings that the micronized carambola IF−vitamin composite was a promising carrier of lipid-soluble bioactive ingredient (i.e., vitamin E) and had slow-releasing characteristics.

According to the National Health and Nutrition Examination Survey III (NHANES III), the vitamin E content of the diets of most of the U.S. population is less than the Recommended Dietary Allowances (12 mg per day for adults) (*26*). Adequate absorption of vitamin E, one of the most important dietary antioxidants, is important for health maintenance such as reducing the risk of cataracts and heart disease and slowing skin aging and anti-inflammation (*27, 28*). Some studies also recommended that dietary supplementation with vitamin E for at least 400 IU (~268 mg) per day was needed to lower the risk of heart attack and protect the body against a variety of types of oxidative stresses (*29*). It was speculated that taking several grams of the above-mentioned MIV composite (10.7 mg of vitamin E g^{-1} of fiber) might help improve the absorption of vitamin E.

After micronization, the particle sizes of carambola IF and cellulose were effectively reduced to micrometer scale with more functional groups being exposed in the fiber matrix. In vitro study demonstrated that micronized insoluble fibers in the forms of micronized fiber−vitamin composites, particularly the MIV-composite, were capable of carrying vitamin E at different extents and releasing the nutrient slowly. Further in vivo evaluation confirmed that the administration of MIV-composite could help maintain the plasma vitamin E at relatively higher levels (about 2.1−3.6-fold of the initial values) for at least 5 h. Our results suggested that micronized insoluble fibers, especially the micronized carambola IF, could be exploited as potential carriers of lipid-soluble bioactive ingredient (i.e., vitamin E) in different food applications and also be used to produce slow-release formulations.

ABBREVIATIONS USED

IF, insoluble fiber; IV, unmicronized carambola IF−vitamin; CV, unmicronized cellulose−vitamin; MIV, micronized carambola IF−vitamin; MCV, micronized cellulose−vitamin; FTIR, Fourier transform infrared spectroscopy; OHC, oil-holding capacity.

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