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Oxidized Tapioca Starch As an Alginate Substitute for Encapsulation of Antioxidant from Coffee Residue

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Abstract

Coffee residue had polyphenol content 18.180 mg/g, yet its extract contained 1.746 mg/g polyhenol. Oxidized tapioca had comparable characteristics to alginate as an encapsulant material. Suspension concentration for encapsulation preparation was 5% (w/v) (encapsulant material/antioxidant extract). Microcapsul which is made by 25% oxidized tapioca starch had loading capacity 33.18%, capsul particle size 1699.3 µm, moisture content 10.57%, polyphenol content 1.23% (db) and antioxidant activity 29.04%. This capsul had better properties than control which is made without oxidized starch substitution.

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Keywords: encapsulation; oxidized starch; alginate substitute; antioxidant

1. Introduction

Indonesia is the biggest fourth coffee producer in the world. Coffee is one of favorite drinks because its unique taste and good health effect for the consumers. Experimental data showed that coffee had high antioxidant activity (Daglia et al., 1994). Other works also showed that roasted coffee took role as an antioxidant and could prevent lipid peroxidation in model system (Stadler et al., 1994). Coffee residue is by product from instant coffee production. Higher demand on instant coffee products produce more waste of coffee residue. We calculate that for instant coffee processing which need 1 kg roasted coffee bean (moisture content 12-13%) resulted 0.743 kg coffee residue

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(moisture content 58.65%) or 0.312 kg (moisture content 4.24%). Yen et al. (2005) reported that coffee residue extract showed high antioxidant activity because it contained polyphenol and non-polyphenol compounds, which called MRPs. This compound can be acted as primary and secondary antioxidant. Products of Maillard Reaction (MRPs), which was produced during roasting process remained in coffee residue.

However, because of the presence of unsaturated bonds in their molecular structure, polyphenols are vulnerable to oxidants, light and heat, which can be easily deteriorated when exposed to these conditions. Therefore, it would be better to protect bayberry polyphenols by some forms from chemical damage by encapsulation. Microencapsulation is described as a technique , for example a bioactive compound, is encapsulated by a biopolymer which can protect the bioactive compound from oxygen, moisture or other stresses to improve its stability (Saénz et al., 2009). Microencapsulation by coacervation is accomplished by phase separation of one or many hydrocolloids from the initial solution and the subsequent deposition of the newly formed coacervate phase around the active ingredient suspended or emulsified in the same reaction media (Gouin, 2004).

Sodium alginate is soluble in water and can form gel beads by dropping an aqueous solution into a divalent or polyvalent cation solu-tion (e.g. Ca2+, Zn2+) to encapsulate active compounds (Draget et al., 1998; George & Abraham, 2006). Although this is a simple and fast way of obtaining encapsulation systems, the method presents a major limitation consisting in loss during bead preparation. Active compound losses are favor by both, the time necessary for the cation to diffuse into the bead and the compound concentration gradient between the beads and surrounding solution. Besides, the presence of macropores in the alginate matrix facilitates the diffusion of hydrophilic molecules (George & Abraham, 2006; Gouin, 2004). However, some researchers were able to solve this problem by mixing alginate with other polymers such as starch, chitosan, cellulose, pectin, among others. In some cases, mechanical and physical properties of beads have been improved, as well (Chan et al., 2011; Santagapita et al., 2012).

Oxidized starch is starch which reacted with oxidizing agent, therefore oxidized starch is less viscous because the starch polymer have been degraded (Bertolini et al., 2001; Dias et al., 2011; Sangseethong et al, 2010; Rivera, 2005) and has higher solubility (Lorlowhakarn et al., 2005 dan Sandhu et al., 2008). These properties promote oxidized starch as suitable candidate for encapsulation matrix. Oxidation product of starch is carboxilate group which has anionic charge (Wurzburg, 1995), so the oxidized starch also makes matrix with divalent or polyvalent cation solution, such as $CaCl_2$. Some oxidized starch have been tried as an encapsulant material, such as oxidized corn starch and oxidised amaranth starch (Kshirsagar, 2008). However application of oxidized tapioca as wall material has not been examined, especially for antioxidant encapsulation of coffee residue extract.

2. 2. Materials and Methods

2.1. Materials

Coffee residue kindly provided by ICCRI Jember, Indonesia (Indonesian Coffee Cacao Research Institution) which is its extract was used as a core. The coating material were combination Alginic acid sodium salt (Sigma) and oxidized stacrh. The oxidized starch was made from commercial tapioca (Brand “99”, Malang, Indonesia) that then was oxidized by hydrogen peroxide. All chemicals used in this study were analytical grade. Purified water was used for the preparation of all solution. All experiments and analysis were carried out in triplicate.

2.2. Preparation of oxidized tapioca

The oxidized tapioca was prepared by following Palupi et al. (2011) method. The tapioca slurry (42g/100ml) was prepared with distilled water and maintained at room temperature. The pH was maintained at 7 by adding NaOH 2N followed with continous stirring for 15 minutes. Hydrogen peroxide was added in the slurry with concentration 1.5% (v/v) accompanied continous stirring for 60 minutes. Then the slurry was sentrifuged to get the starch and the supernatant was decanted. The oxidized starch was dried in convection oven at 50°C for 20 h then was kept in sealed box at room temperature.

2.3. Extraction of polyphenol from coffee residue

Coffee residue was dried in convenction oven at 50C for 48 h and its moisture content became 4%. Dried coffee residue was extracted with double solvents. First, it was extracted with alcohol 96% and ratio of sample: alcohol was 1 : 8. The extraction was followed by continous strirring for 30 minutes then maseration for 4 h. Filtrate was collected and its residue continued for second extraction with distilled water (residue:aquades = 1:8). In this extraction stirring was run for 30 minutes and maseration for 17 h . The filtrate which was resulted from second extraction was mixed with previous extract. Finally, the filtrate evaporated by roraty evaporator at 40C until its volume became about one fourth from previous volume .

2.4. Encapsulation by coacervation

Suspension for encapsulation were made in concentration 5% and 10% (w/v). Encapsulation matrix was composed by 3% oxidized tapioca and sodium alginte, and the rest of 2% was sodium caseinate. Concentrations of oxidized tapioca to substitute alginate were 25% and 50% (Table 1). The matrix was dissolved into 100 ml evaporated coffee residue extract which was prepared in beaker glass 250 ml. The suspension was placed on hot plate and was kept at the same temperature. This process was followed by continous stirring in order to maintain homogenous suspension. Once it was homogenize, the solution was forced into a syringe (diamtere 1 mm) to drop into a calcium chloride solution ($CaCl_2$ 0.1 M). The The beads were maintained in the gelling bath to harden for 15 min. The they filtered and washed with distilled water. These hydrogels beads containing coffee residue extract will be referred as A, B, C, D, and E. These beads then were dried at room temperature.

Table 1. Composition material encapsulation

Sample code	Capsule suspension (%)	Oxidized tapioca concentration (%)	Alginate concentration (%)
A	5	0	100
B	5	25	75
C	5	50	50
D	10	25	75
E	10	50	50

2.5. Polyphenol content

Total polyphenol content was determined by the Folin-Ciocalteu method (Schlesier et al., 2002). Briefly, 2 mL of Na_2CO_3 (2 g/100 mL) (Sigma-Aldrich) were mixed with 200 L of the sample and 200 L of Folin-Ciocalteau reagent. After 30 min, sample absorbance was measured at 725 nm in a spectrophotometer (Shimadzu, UV-mini 1240, Japan). Results were expressed as mg standard equivalent/g of sample.

2.6. Scavenging effect on DPPH radical

The scavenging effect on DPPH radicals was estimated according to the procedure described by Parejo et al. (2000) and Chen & Xu (2007), using slight modifications. Diluted sample solutions (0.1–6 mg of samples/ml of ethanol) were prepared for determination. Each diluted solution (0.1 ml) was added to 3.9 ml of a 6.0 x 10-5 M ethanol solution of DPPH. The reaction was allowed to take place in the dark at room temperature to reach steady state conditions, after which time the decrease in the absorbance was determined at 515 nm. Ethanol was used to zero spectrophotometer.

2.7. Particle size and its visual appearance

Diameter of the capsules were examined by Optilab Microscope Olympus. Then in order to get visual perception on capsules size, the capsules compared to small tablet which had been known its diameter. The pictures were recorded by camera Nikon coolpix.

2.8 Data Analysis

All experiments were carried out triplicate and the results were reported as averages and standard deviations of these measurements.

3. 3. Results and discussion

3.1. Characteristics of coffee residue extract

Table 2. Polyphenol content and antioxidant activity of coffee residue and its extract

Parameters	Dried coffee residue	Coffee residue extract
Moisture content	4.2 ± 0.21	91.91 ± 0.75
Polyphenol content (mg/g)	18.18 ± 0.31	17.75 ± 0.06
Polyphenol content (%db)	3.88 ± 0.018	4.49 ± 0.24
Antioxidant activity (% DPPH)	16.01 ± 0.55	62.81 ± 1.03

The extract was prepared from dried coffee residue then both were examined its moisture content, its polyphenol content (mg/g and %db) ,and its scavenging effect on DPPH radical. The polyphenol content (%db) was calculated by considering its moisture content. For the next discussion, when we discuss about polyphenol content, it is %db of polyphenols of samples.

The data (Table 2) showed that polyphenol content (%db) and antioxidant activity of coffee residue is lower than its extract. It perhaps in the extract, polyphenols compound were not complexed with other compounds.

3.2. Role of combination coating materials on capsule characteristics

Tabel 3. Capsule characteristics

Capsule	Moisture content (%)	Polyphenol content (%db)	Antioxidant activity (% DPPH)	Particle size (μm)
A	12.63 ± 0.12	1.28 ± 0.032	27.84 ± 0.87	1732.7 ± 171.5
B	10.57 ± 0.30	1.23 ± 0.059	29.04 ± 0.24	1699.3 ± 75.60
C	11.07 ± 0.39	0.95 ± 0.032	18.70 ± 0.73	1633.7 ± 31.78
D	12.71 ± 0.07	0.86 ± 0.024	14.00 ± 0.061	2194.5 ± 189.05
E	13.63 ± 0.40	0.83 ± 0.041	18.50 ± 0.77	1735.8 ± 66.40

Different capsule suspensions affected different moisture content characteristic of the capsules. Alginate-oxidized tapioca capsules (B and C) which were made in 5% suspension had lower moisture content than alginate capsule which was produced in similar suspension concentration (A). Otherwise, alginate-oxidized tapioca capsules (D and E), which were resulted by suspension 10% had higher moisture content than alginate capsule prepared by 5% suspension (A).

Nonetheless, alginate capsules could not be produced in 10% suspension because its viscous suspension could not drop from the syringe which had small diameter (0.1 mm), so we did not compare the capsules with alginate capsule prepared by 10% suspension, which were composed by alginate and oxidized tapioca were lower than

alginate capsule. However, both capsules of 5% suspension (B and C) and capsules of 10% suspension (D and E), The data of moisture content in this study were similar with Donithi et al. (2011) work which reported moisture content of freeze dried capsules were about 11.11% – 16.61%. Capsules in their work had matrix composition lecithin-starch-alginate.

4. 4. Conclusions

Oxidized tapioca has potency as an alginate subtitue for encapsulant material. Microcapsul which is made by 25% oxidized tapioca starch has better properties than alginate capsule with loading capacity 33.18%, capsul particle size 1699.3 µm, moisture content 10.57%, polyphenol content 1.23% (db) and antioxidant activity 29.04%.

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