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Utilization of Carboxymethyl Cellulose (CMC) from Coconut Coir Waste (*Cocos nucifera*) as a Stabilizer in Red Bean (*Phaseolus vulgaris* L) Vegetable Milk

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ABSTRACT

Carboxymethyl cellulose (CMC) synthesis from coconut coir waste provides an alternative that can be used as a stabilizer to manufacture red bean milk. The first step is to isolate alpha-cellulose with a 2% NaOH solution. Second, the etherification process is alkalization with isopropanol and 17.5% NaOH solution to produce alkaline cellulose, and then the carboxymethylation process with sodium monochloride acetate (NaMCA) and characterized by FT-IR. The resulting carboxymethyl cellulose is purified by centrifugation. Red bean milk was analyzed using proximate analysis, viscosity, percent stability, pH, and organoleptic content by varying CMC 0.1, 0.2, and 0.5%. The alpha cellulose obtained was 13.37%, and the CMC produced from the etherification and purification process was 5.97 g. The characterization of alpha-cellulose showed (OH) at wave number 3441cm$^{-1,}$ and the CMC characterization showed carbonyl (COO-) at wave number 1604.77 cm^{-1} and group ether (CH_2-O-CH_2) at wave number 1419.77. Proximate analysis of red bean milk protein content of 2.75%, carbohydrates 10.27%, fiber 8.5%, and fat 18.57% and results based on viscosity and percent stability were obtained by adding 0.5% CMC and organoleptic test results adding CMC preferably with 0.3%, the texture is not too thick and even, and the aroma of red beans is typical.

Keywords: Alkalizations, Carboxymethyl Cellulose, Red Bean, Viscosity

ABSTRAK

Sintesis karboksimetil selulosa (CMC) dari limbah sabut kelapa memberikan alternatif yang dapat digunakan sebagai bahan penstabil dalam pembuatan susu kacang merah. Langkah pertama mengisolasi alfa selulosa dengan larutan NaOH 2%. Kedua, proses eterifikasi adalah alkalisasi dengan larutan isopropanol dan NaOH 17,5% untuk menghasilkan selulosa alkali dan selanjutnya proses karboksimetilasi dengan natrium monokloro asetat (NaMCA) dan dikarakterisasi dengan FT-IR. Hasil karboksimetil selulosa dimurnikan dengan sentrifugasi. Pembuatan susu kacang merah dengan parameter, analisis proksimat, viskositas, persen stabilitas, pH dan kandungan organoleptik dengan memvariasikan CMC 0,1 0,2 dan 0,5 %. Hasil isolasi alfa selulosa diperoleh 13,37% dan CMC yang dihasilkan dari proses eterifikasi dan pemurnian adalah 5,97 g dan karakterisasi alfa selulosa menunjukkan (OH) pada bilangan gelombang 3441 dan karakterisasi CMC menunjukkan karbonil (COO-) pada bilangan gelombang 1604,77 dan eter gugus (CH_2-O-CH_2) pada bilangan gelombang 1419.77. Analisis proksimat kadar protein susu kacang merah 2,75%, karbohidrat 10,27%, serat 8,5% dan lemak 18,57% serta hasil berdasarkan viskositas dan persen stabilitas diperoleh dengan penambahan CMC 0,5% dan hasil uji organoleptik penambahan CMC disukai dengan 0,3% teksturnya tidak terlalu kental dan merata serta aroma kacang merah yang khas.

Keyword: Alkalisasi, Kacang merah, Karboksimetil Selulosa, Viskositas

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1. Introduction

In the trading world, coconut coir is known as coco-fiber, coir fiber, coir yarn, coir mats, and rugs [1]. The main content of coconut coir is 22% cellulose, 10% hemicellulose, 47% lignin, 12% water, 1.5 % ash, and 7.5% extract [2]. The high content of cellulose in coconut coir has the potential to be processed into cellulose derivatives, one of which is Carboxymethyl Cellulose (CMC). Carboxymethyl Cellulose (CMC) is a cellulose derivative prepared by exchanging hydroxyl groups of cellulose with carboxyl groups contained in monochloroacetic acid under alkaline conditions. Carboxymethyl cellulose is a linear cellulose polymer ether and is an anionic compound that is biodegradable, colorless, odorless, and non-toxic [3]. CMC is widely used in the pharmaceutical, food, textile, detergent, and cosmetic product industries [4]. CMC also functions to maintain the stability of the drink so that the solid particles are dispersed evenly throughout so that they do not experience precipitation [5]

One of the vegetable milk that is widely known by the public is soy milk. Soy milk is a beverage product that is the same as cow's milk but made from soybeans, so it can be consumed by those who are allergic to cow's milk. It's just that the costly price of soybeans makes people start looking for alternative types of beans that can be made with milk, one of which is tolo beans and red [6].

People consume red beans (*Phaseolus vulgaris* L.) worldwide; many countries, such as South America, Asia, and Africa, use them as the main staple food. Nuts are a good source of protein, energy, vitamins, fiber, and minerals and are saturated fat-free [7]. The great benefit of red beans can be used as processed food or beverage products that attract consumers. Red bean processing into beverage products can be an alternative to consuming vegetable milk other than soy and milk derived from animal products.

Precipitation in the juice or milk in the nuts can affect the quality and level of consumer preference. Therefore, the stability of the drink's suspension from the nuts' juice must be maintained to prevent precipitation. A stabilizer can be added. One of the stabilizers that can be used is CMC [8].

2. Materials and Methods

2.1. Equipment

The tools used in this study were Erlenmeyer, beaker glass, aquadest, measuring flask, measuring cup, centrifugation, oven, analytical balance, desiccator, FTIR, burette, Ostwald's viscometer, Kjedhal flask, condenser, pycnometer, electrode pH meter, soxhlet flask.

2.2. Materials

The materials used in this study were coconut fiber, nitrate acid (HNO_3) 3.5%, Sodium nitrite ($NaNO_2$), hydrochloric acid (HCl) 0.1 N, hydrochloric acid (HCl) 3%, sodium hydroxide (NaOH), sodium hydroxide (NaOH) 17.5%, sodium sulfite (Na_2SO_3) 2%, hydrogen peroxide (H_2O_2) 10 %, ethanol 70%, methanol, isopropanol, sodium monocloroacetate (NaMCA), acetic acid 90%, aquades, acetone, copper (II) sulfate ($CuSO_4.5H_2O$), 1-Naptol, hydrochloric acid (HCl) 37%, sulfuric acid (H_2SO_4) 10%, sulfuric acid (H_2SO_4), sulfuric acid (H_2SO_4)1.25%, *n*-Hexane, Luff Scroll Solution, boric acid (H_3BO_3) 3%, ethanol 96%, Selenium mix, sodium hypochlorite (NaOCl) 1.75%, Tashiro Indicator, P.P. Indicator, amilium indicator, Iodine 0.01N, 10% K.I. Red Beans and sugar.

2.3. Provision of Samples

Coconut coir was taken around Tanjung Sari Medan's Melati tax, then dried under the sun to dry. After that, the coconut fiber is cut into small pieces and mashed using a blender.

2.4. Isolation of α–Cellulose from Powder Coconut Coir

In order to isolate the α–Cellulose, 75 g of the coconut coir powder was weighed into a beaker glass, then add 1000 mL of 3.5% (v/v) HNO_3 and 10 mg of $NaNO_2$ heated at 90˚C for 2 hours, stirring over a hot plate. Filtered, and the residue was washed until the filtrate was neutral, added 375 mL NaOH 2% (w/v) and 375 mL Na_2SO_3 2 % (w/v) were heated at 50ºC for 1 hour, filtered, and washed the filtrate until neutral, bleached with 500 mL of 1.75 % (v/v) NaOCl solution, heated at 70ºC on a hot plate, filtered and cleaned until the filtrate is neutral and added 500 mL of 17.5% (w/v) NaOH and heated to 80 ^{0}C for 30 minutes. The residue was washed until the filtrate was neutral, added 250 mL H_2O_2 10 % (v/v) was heated at 60ºC for 15 minutes, filtered, and washed with distilled water until the filtrate was neutral, dried in an oven at 60ºC and stored in a desiccator and characterized by FT-IR spectroscopy[9].

2.5. *Synthesis of Carboxymethyl Cellulose (CMC)*

Synthesis of Carboxymethyl Cellulose (CMC) was conducted by weighing 5g of α-cellulose and put into a 250 mL beaker glass, adding 100 mL of isopropanol, adding 20 mL of 17.5% (w/v) NaOH solution, slowly stirring for 1 hour at 30°C. Add 6g of sodium monochloro acetate (NaMCA). The mixture was placed in a water bath, heated at 50°C, and shaken for 2 hours. Soaked in methanol for one night, neutralized with CH_3COOH 90% to pH 6-8, and filtered. The residue was washed with 70% ethanol five times, filtered, dried in an oven at 60°C for 24 hours, and stored in a desiccator [10].

2.6. Purification of Carboxymethyl cellulose (CMC)

Put 5 g of CMC into a 250 mL beaker glass, dissolve with 100 mL of distilled water, heat on a hot plate at 80^0 C for 10 minutes, and stir. Then centrifuged for 1 minute at 4000 rpm, the precipitate was separated from the solution. Dissolve the reprecipitated CMC with 100 mL of acetone and filter. The residue is wrapped in aluminum foil, dried in an oven at 600C for 4 hours, stored in a desiccator, and characterized using FT-IR [11].

2.7. Making Red Bean Milk

Red beans were sorted, washed with clean water, soaked in water for 17 hours, and heated at 80°C for 15 minutes. Then the red beans were mashed using a blender and water at 40ºC, filtered, and the filtrate and residue were produced. 100 mL of red bean filtrate was added with CMC 0.1; 0.3; 0.5%, and sugar, then pasteurized at 60°C [12].

2.8. Protein Level Test

As much as 1g of red bean vegetable milk into the Kjhedal flask, add 2 g of selenium mix and 15 mL of H_2SO_4, and heat it over the Kjhedal apparatus until the solution is clear. Cooled and put into the bottom flask and added 100 mL of distilled water, 30% NaOH (w/v), and distilled for a few minutes. The distillate results were collected in an Erlenmeyer containing 3% (v/v) H_3BO_3, and added the Tashiro indicator until the color changed to green. The distillate results were then titrated with standard 0.1 N HCl solution until the solution changed color from green to purple, the volume of titrant used was recorded, and the protein content was calculated.

2.9. Fat Content Test

Two grams of red bean vegetable milk were weighed and placed into a beaker glass, added 30 mL of 25% HCl (aq) and 20 ml of distilled water, boiled for 15 minutes, then filtered and washed with hot distilled water until it no longer acts sour. The filter paper and its contents are dried at 100-105°C. The filter paper is wrapped and put into the soxhlet apparatus. Extracted with n-Hexane solution for 2-3 hours at 80 0 C. Distilled n-Hexane solution from fat extract at 100-105°C. Cooled in a desiccator and weighed to constant weight.

2.10 . Carbohydrate Level Test

2 g of sample was put into the beaker glass, add 50 mL of distilled water, add 25 mL of 3% HCl (v/v), and 1 mL of 0.01N iodine, heated to boiling, and pipette 10 mL of the solution, put it into an Erlenmeyer containing 25 mL of Luff Schrool solution and 15 mL of distilled water. Heated to boiling and added 15 mL of 10% K.I. and 10% (v/v) H_2SO_4. Titrate with 0.2N Na_2SO_3 solution, add three drops of starch indicator, titrate until the solution is milky white, and record the titrant volume.

2.11 . Fiber Content Test

2 g red bean vegetable milk was added 50 mL of H_2SO_4 1.25 % (v/v) and boiled for 30 minutes added 50 mL NaOH 3.25% (w/v) boiled for 30 minutes, filtered through Whatman filter paper No. 41, washed with H_2SO_4 1.25% (v/v) which has been heated and washed with hot distilled water and 96% (v/v) ethanol put into a cup whose weight is known to be dried in an oven at 105^0 C cooled in a desiccator and weighed to constant weight and calculated fiber content.

3. Results and Discussion

3.1. Results of Isolation of Cellulose and Synthesis of CMC from Coconut Coir

Isolation of cellulose takes two steps, delignification and bleaching stages. Synthesis of carboxymethylation takes place in two stages, alkalization, and carboxymethylation (etherification).

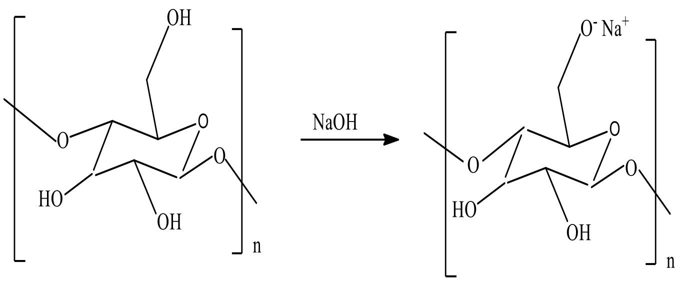

Figure 1. Cellulose Alkalization reaction (Alfian, Hadi.2014)

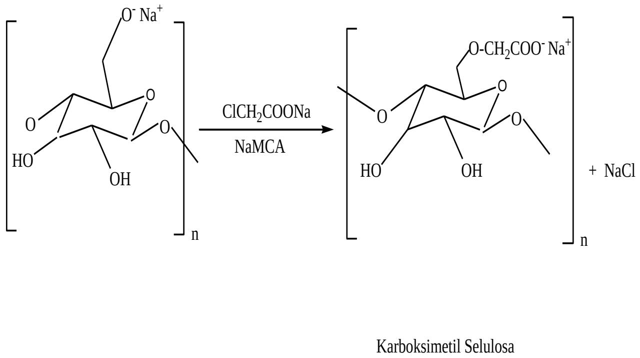

Figure 2. Carboxymethyl cellulose reaction mechanism [12]

3.2. Results of Cellulose and CMC FT- I.R. Spectrophotometer Analysis

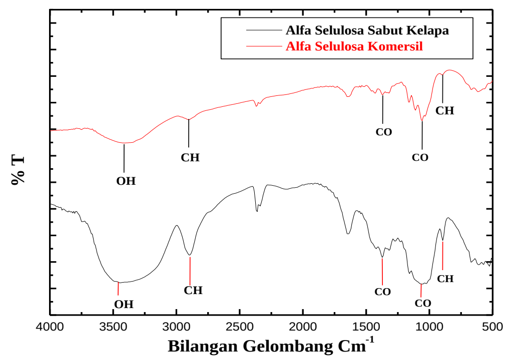

Figure 3 FTIR spectrum of alpha coco cellulose and commercial alpha-cellulose.

The FT-IR spectrum shows the emergence of wave numbers in the region 3441 cm^{-1} and 3410 cm^{-1} is a hydroxyl group (-OH), and 1643.35 cm^{-1} is a carbonyl group (C=0). Wave numbers 2893^{-1} and 2900 cm^{-1} contain C.H. groups from the alkane chain [13]. The wave numbers in the areas 1373 and 1319 cm^{-1} and in the areas 1026 cm^{-1} and 1056 cm^{-1} indicate the presence of C.O. strain in cellulose [14,15]. Meanwhile, the C.H. swing vibration was found in the absorption area of 894 cm^{-1} in α-coconut coir cellulose and commercial α-cellulose, indicating the presence of β-glycoside bonds in these structures [16].

3.3. Results of FT-IR Spectrophotometer Analysis of Coconut Coir CMC and Commercial CMC

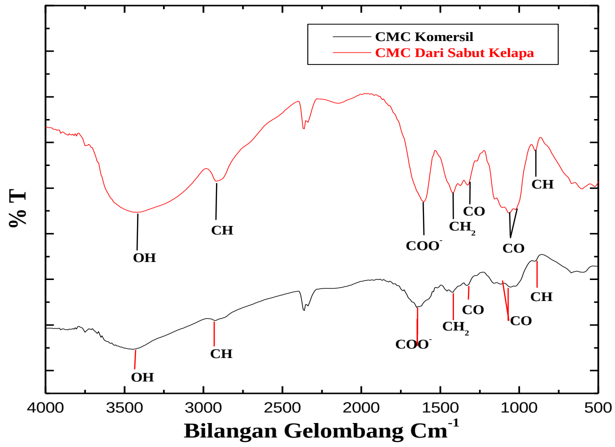

Figure 4. FTIR spectrum of coconut coir CMC and commercial CMC

The FT-IR spectrum of carboxymethyl cellulose from coconut coir and commercial carboxymethyl cellulose shows that the wave numbers in the absorption areas 3425 and 3448 cm-1 are the stretching of the -OH group and the appearance of vibrations in the absorption waves 2919 and 2924 is the C.H. group [17]. The carboxyl group and its salt show two absorption peaks at wave numbers between 1600-1640 cm -1 and 1400-1450 cm-1, carboxymethyl substituents (COO-) and -CH2 bonds. This is a characteristic of carboxymethyl cellulose compounds [18]. According to [19], CMC was identified as having a carboxyl group (COO -) at a wavelength of 1064 cm-1 and at a wavelength of 1419 cm^{-1} which is a $–CH_2$ bond, from the results of the analysis, carboxymethyl cellulose from coconut coir and commercial carboxymethyl cellulose have similarities and are carboxymethyl cellulose compounds.

3.4. Proximate Test Results (Protein, Fat, Fiber, and Carbohydrate)

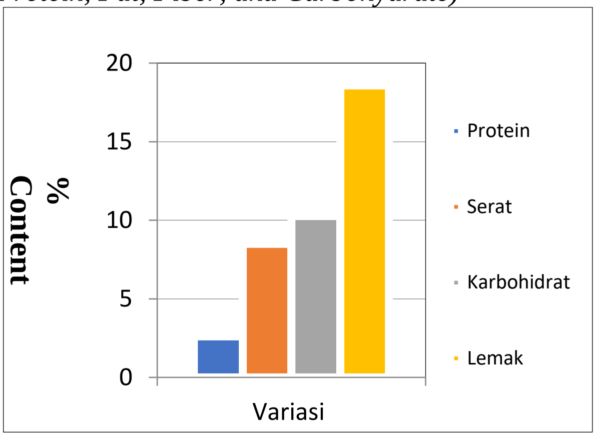

Figure 5. Red Bean Vegetable Milk Proximate Test

The raw materials used in production significantly affect the product characteristics' value. Protein, fat, carbohydrates, and fiber are essential in a beverage or food product. The methods used in the proximate analysis are the Kjhedal method, Soxhlettation, Frude Fiber, and the Luff Scroll method. The analysis found that the protein content of red bean vegetable milk was 2.75%, fat was 18.57%, fiber was 8.5%, and carbohydrates were 10.275%. The proximate analysis results of red bean vegetable milk were where the percentage of protein still meets the standardization of soybeans [20], where the percentage of protein in drinks is 1%.

3.5. Percent Stability Test Results for Red Bean Vegetable Milk

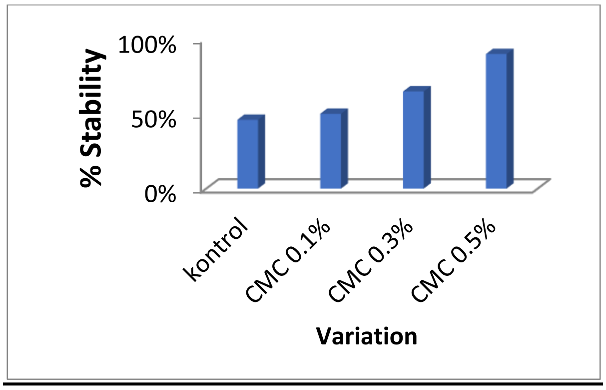

Figure 6. Bar chart of the effect of adding CMC concentration on the stability of red bean vegetable milk.

The results of the analysis of the percent stability of red bean vegetable milk showed that there was an effect of the process of adding CMC. Where from the results of adding 0.5% CMC was very influential on the percent stability. This is because CMC has anionic properties that cause interactions with many proteins and form soluble and stable complexes [21]. The low percent stability in the control treatment was due to the absence of CMC addition treatment, so all suspended particles in the red bean vegetable milk also precipitated.

3.6. Results of pH Measurement (Acidity Degree)

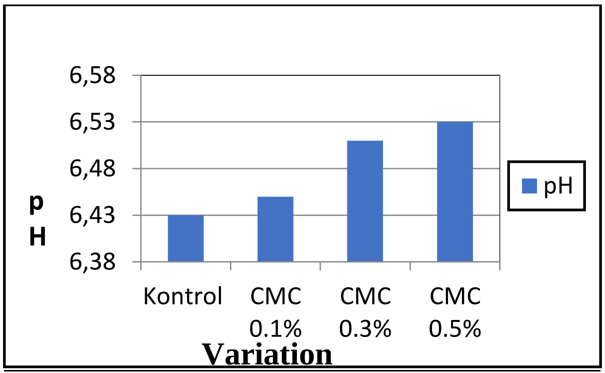

Figure 7. Bar diagram of the effect of adding CMC to the pH of red bean vegetable milk.

The analysis results showed that the difference was not that big between the treatments of adding CMC to the pH value of the red bean vegetable milk drink. Adding 0.5% CMC resulted in a higher pH than adding 0.1%, 0.3% CMC, and control. It was due to CMC having hydrocolloid properties containing many carbonyls so that it hydrolyzed easily, and this was supported by [22] the addition of CMC in higher concentrations can cause an increase in pH in food and beverage products. This is because CMC has a carboxyl group. From the analysis results, the pH value of red bean vegetable milk ranges from 6-6.5. This is still following the standardization of soybean drinks (SNI 01-3830-1995).

3.7. Viscosity Test Results

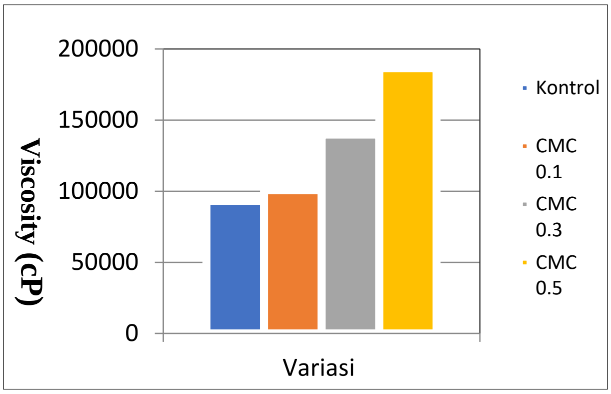

Figure 8. Bar diagram of the effect of adding CMC concentration on the viscosity of red bean vegetable milk.

Figure 8 shows that the analysis results show that adding CMC affects the red bean vegetable milk solution. It is due to the greater concentration of CMC added as a stabilizer, the greater the amount of water bound by CMC, so that the gel state and the molecular weight value of the viscosity of the drink red bean vegetable milk will increase. This is supported by [23], which states that an increase in the concentration of CMC in the solution can result in a large amount of bound water, the more significant the increase in viscosity. In contrast, the lowest viscosity value is obtained in the control variation. It is due to the absence of the addition of a stabilizer that can bind water, so the viscosity is very low.

3.8. Organoleptic Test

3.8.1. Texture

The texture is an important aspect of food quality; texture can also affect food taste. The analysis results showed that the different levels of CMC addition affected the quality of the red bean vegetable milk drink. Where the panelists preferred variations of the addition of 0.1% and 0.3% CMC while the panelists did not like the 0.5% CMC concentration variations, this was because the texture was very thick, which reduced the panelists' preference value. According to [24], the mechanism of action of CMC as an emulsion stabilizer is closely related to its very high ability to bind water, thus increasing the viscosity of the solution. The more CMC added, the lower the preference level of the panelists for the drink.

3.8.2. Scent

Aroma is one factor that significantly influences the value of the quality of a food or beverage product. The survey results show that the quality of the aroma of red bean milk does not affect the addition of CMC concentration. It occurs because the nature of CMC is odorless, non-toxic, and has no aroma.

3.8.2. Flavort

Taste is one factor that significantly influences the quality of a food or beverage product. From the survey that has been carried out, the organoleptic value of taste shows that the panelists like the sweet taste of red bean vegetable milk due to the addition of the same concentration of sugar to the red bean vegetable milk. This shows that the addition of the attention of the stabilizer does not affect the taste value of the drink because CMC is a substance with a white or slightly yellowish color, odorless and tasteless [25].

4. Conclusion

The alpha-cellulose isolated results were obtained at 13.37%. The CMC produced from the etherification and purification process was 5.97 g and the characterization of alpha cellulose showed (OH) at wave number 3441 cm^{-1}. The CMC characterization showed carbonyl (COO-) at wave number 1604.77 and group ether (CH_2-O-CH_2) at wave 1419.77 cm^{-1}. Proximate analysis of red bean milk protein content of 2.75%, carbohydrates 10.27%, fiber 8.5%, and fat 18.57% and results based on viscosity and percent stability were obtained by adding 0.5% CMC and organoleptic test results adding CMC preferably with 0.3%, the texture is not too thick and even, and the aroma of red beans is typical. Based on the research, it can be concluded that the concentration of 0.5% CMC is the best stabilizer and has the best percent stability, and the addition of 0.3 % CMC, the concentration of organoleptic values preferred by panelists.

5. Acknowledgements

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6. Conflict of Interest

The authors declare no conflicts of interest.

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