6th iSNPiNSA

International Seminar on New Paradigm and Innovation on Natural Sciences and Its Application

"SCIENCE AND ITS APPLICATION FOR PRODUCTIVE AND SUSTAINABLE DEVELOPMENT"

Semarang, October 5-6, 2016
Grand Candi Hotel
Jl. Sisingamangaraja, Semarang, Indonesia

PROCEEDING

RESEARCH PAPER IN BIOLOGY AND CHEMISTRY

Part 2 of 2 (Page: 285 – 679)

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DIPONEGORO UNIVERSITY
SEMARANG

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The Optimization of Cream Formula Nanoparticles of Corncob Waste (*Zea mays L.*) Ethanol Extract Potential as Sunscreen

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The antioxidant effect of phenolic compounds contained in the ethanol extract of corncob waste due to oxidizing characters thats play a role in neutralizing free radicals as well as a sunscreen. This study aims at acknowledging the optimum formula of the corncobs ethanol extract as the utilization of corncob which is a waste into a sunscreen active ingredient preparation in the form of cream with the reduction of particle size of the corncobs ethanol extract. The preparation optimization parameter includes preparation physical stability test such as preparation pH test, preparation cream type, dispersive power capability and preparation adhesive power. The optimization calculation by using design software expet 9. Besides, there has also conducted an establishment of total phenolic degree of corncobs extract nanoparticles and cream preparations of corncobs extract nanoparticles.

In particle size test, all of the tested formula are still in microparticles (over 100 nm). This indicates the need for optimization of various extracts addition, chitosan and Na-TPP to produce nanoparticles and need to try to use other crosslinking such as pectin and alginate. The ethanol extract of corncob particles coated chitosan to be added in the cream is the ethanol extract of corncob with level of 6 mg/ mL with chitosan ratio 1:1, because it has smallest average particle size.

The optimum formula both of cream base or cream preparation with active ingredients of corncobs ethanol extract microparticles are creams that contains tween comparison 80 and span 80 at 15: 1 with the observation result of each parameter proved not to significantly differ from the results of software prediction of design software expert 9. The mean levels of total phenolic determination result in the corncobs ethanol extract is 48.4685 ppm and the cream preparation of corncobs extract nanoparticles is obtained 189.8923 ppm.

Keywords : Optimization, Cream, Nanoparticles, Corncob waste, Sunscreen

1. Introduction

Sun light, on one hand is really needed by living creatures as an energy source and skin and bones nutrient, but on the other hand, sunlight contains ultraviolet rays that can harm the skin. The intensity of highest UV radiation is at 08.00 - 15.00 local time, when people are active outdoors (Iswari, et al, 2007). One way that can be done to minimize the amount of UV that penetrates into the skin is to use sunscreen. Indonesia as a tropical country with high sun exposure really needs cosmetics preparation that acts as a sunscreen.

The phenolic compounds may act as a sunscreen to prevent any harmful effects of UV radiation on the skin due to its antioxidant as photoprotective (Svobodova et al., 2003). This matter is supported by Panovska, et al. (2005) that stated the antioxidant compound is an inhibitor used to inhibit the autooxidation. The antioxidant Effect of phenolic compounds due to oxidizing characters that play role in neutralizing free radicals.

Saleh, et al. (2012) stated that the active compounds contained in extracts of corncobs have a good ability to counteract free radicals that works to inhibit oxidation by reacting with reactive

free radicals to form unreactive free radicals that is relatively stable, thus enables beneficial in slowing the process of photooxidation due to exposure UV sun light. Besides, in a study (Wungkana, et al, 2013) showed that the phenolic content of ethyl acetate fraction of corncobs waste can act as an antioxidant as well as a sunscreen.

This study aims at ackowledging the optimum formula of the ethanol extract of corncobs as the utilization of corncobs which is waste to become active ingredient preparation of sunscreen in the form of cream with the reduction of particle size of the corncobs ethanol extract . The preparation optimization parameters include preparation physical stability test such as preparation pH test, preparation cream type, dispersive power capability and preparation adhesiveness.

2. Research Method

Raw materials used in this study is hybrid corncobs waste from Karangawem region, Mranggen subdistrict, Demak Regency. The chemicals substance used are ethanol, natrium carbonate, natrium lauryl sulphate, cetostearyl alcohol, stearic acid, TEA, lanolin, Span 80, VCO (Virgin Coconut Oil), propylene glycol, methyl paraben, propyl paraben, aquadest.

The equipments used are erlenmeyer, separating funnel, transparent glass bottles, micropipette, test tubes, reflux tools (electric heater, glass pumpkin , condensers), vortex, electric balance, rotary evaporator, spectrophotometer ,UV-Vis, mortar grinders, oven, bath sonicator , analytical balance, brookfield viscometer, pH meter, water bath.

Sample Preparation

The dried corncobs are cut into small pieces and destroyed by means of blender.

Sample Extraction

The Corncobs were extracted by using ethanol solvent 70%. Extraction is conducted by reflux. A total of 250g of corncobs inserted into the distillate flask, 1500 mL ethanol was added to the sample to submerged, then heated for 2 hours at a temperature of 78-90°C. The filtrate was filtered and then evaporated to remove the solvent by using a rotary evaporator, then dried up to dryness to obtain a crude extract of corncobs. The extract was fractionated by four solvents which are n-hexane, ethyl acetate, water.

The determination of total Phenolic Content

The sample of ethanol extract and corncobs fraction is then determined the total phenolic content by using the Folin-Ciocalteu method. A total of 0.1 mL of each fraction of 1000 ppm inserted into a test tube, then added 0.1 mL of Folin-Ciocalteu reagent 50%. The mixture then divortexed for 2 minutes, then added 2 mL of 2% sodium carbonate solution. After that, the mixture was incubated in the dark room for 30 minutes.The absorbance is read at λ 750 nm by using a spectrophotometer. The total content of phenols was stated as gallic acid equivalents mg / kg extract.

The fraction with the highest total phenolic content will be used in the next stage. In the previous study (Wungkana, et al., 2013) was known that the highest total phenolic content contained in ethyl acetate fraction.

The manufacturing of ethanol extract particles of chitosan coated corncobs waste

The extract solution was made by several concentrations of the extract, i.e ranging from 1 mg/mL, 1.5 mg/mL, 3 mg/mL, 6 mg/mL, up to 9 mg/mL. Each extract with those levels was made in 70% ethanol solvent.

The chitosan solution with concentration of 3 mg/mL was made by dissolving powdered chitosan within acetate buffer solution pH 4 and stirring with a magnetic stirrer until the chitosan is

dissolved. The solution was filtered to avoid other particles. Solution of Na-TPP as a crosslink was made by a concentration of 3 mg/mL in aquadest solvent.

The formula of ethanol extract particles of chitosan coated corncobs waste are made by:

1) The extract solution was inserted into the vial, then added by a solution of chitosan and divortex for 30 seconds.
2) Na-TPP as much as 0.1 part of the total volume was added and divortexed back for 30 seconds.
3) The Formula of optimum chitosan coated particles indicated by no occurrence of precipitation during 3 days storage.
4) As for some formulas of chitosan and extracts comparison were made to obtain the coated particles of the best chitosan.

The instruction of how to make cream preparation on base 1 optimization are as follows :

1) Stearic acid, white vaseline, cera alba and span 80 are melted in a porcelain dish above a water bath (mixture 1).
2) TEA and tween 80 are melted in other porcelain dish above a water bath (mixture 2)
3) A Mortar prepared with hot temperatures.
4) Mixture 1 is poured into a hot mortar, then added VCO little by little, stir it until homogeneous (mixture 4).
5) Mixture 2 is poured into mixture little by little while keep stirring until homogeneous (mixture 5).
6) The microparticles of ethanol extract of corncobs and aquadest are added into mixture 5 little by little while keep stirring until homogeneous and creamy mass is formed.
7) The cream is poured into cream pot that has already prepared
8) Conducting evaluation test of cream preparation physical characteristics.

The physical cream evaluation

Organoleptic

Cream preparation observed occurrence of phase separation or breaking up of the cream preparation.

pH measurement

pH measurement was conducted by using a pH-meter. At first, the electrode was calibrated with standard buffer of pH 4 and pH 7. Then, the electrode was dipped into the preparation. The pH value displayed in monitor then recorded. Measurement was conducted at room temperature.

Cream type checking

The Cream type determination was conducted by using coloring techniques. Three drops of methylene blue is dripped into 3 drops of cream, and then observed by using a microscope. If the emulsion cream has the same color then the tested cream is manifold as m / a (Ansel, 1989).

Table 1. The optimation of cream preparation formula with active ingredients of corncobs waste ethanol extract nanoparticles.

Ingredients	Formula (in one gram)						
	I	II	III	IV	V	VI	VII
Microparticles of corb cobs ethanol extract	7,5	7,5	7,5	7,5	7,5	7,5	7,5
Stearic acid	22,5	22,5	22,5	22,5	22,5	22,5	22,5
TEA	2,25	2,25	2,25	2,25	2,25	2,25	2,25
Tween 80	15	1,5	8,25	4,875	11,625	1,5	15

Span 80	1,5	15	8,25	11,625	4,875	15	1,5
Cera alba	3	3	3	3	3	3	3
White Vaselin	12	12	12	12	12	12	12
Virgin Coconut Oil (VCO)	12	12	12	12	12	12	12
Aquadest	ad 150	ad 150	ad 150	ad 150	ad 150	ad 150	ad 150

Dispersive Power Test

A total of 0.5 g cream is put in the middle of tool with diameter of 15 cm which one glass placed upon and left for 1 minute. Measure the diameter of the spreading preparation cream, then added 50 g of additional weight and let it for 1 minute and measure the diameter of spreading calamine. This is conducted repeatedly until we get constant spread diameter. Constan. Replication is conducted 3 times.

Adhesiveness Test

1 mg Cream Preparation is taken then smeared on a glass plate, affixed to both plate to unite the plates then press with a weight of 1 kg for 5 minutes after that release release the weight, then give a release wiehgt 80 r for testing. Record the time until both plates are separated.Conduct replication for 3 times.

3. Result and Discussion

Corncobs waste from Karangawen-Mranggen region, Demak then extracted with 70% of ethanol through the reflux method. 400 grams of powder corncobs waste extracted with ethanol 70% as much as 4 L through reflux method .The yield obtained from the extraction is 2.70%. Total phenolic content in each extracts is stated as gallic acid equivalents. The ethanol extract of the corncobs have a total phenolic content of 7.34% or 73.4 mg per kg extract.

The yield which slightly encourages the researchers to try using p.a ethanol solvent as a solvent extraction of corncob waste. 150 grams of powder corncob waste is extracted with p.a ethanol solvent as much as 500 mL through reflux method. The reflux Result then evaporated by using a rotary evaporator until obtaining dry extract. But, the results of the yield is not much different from the extraction that uses 70% ethanol, so, to make chitosan coated extract particles keep using ethanol obtained by extraction that uses 70% ethanol.

The expected mixture of extracts and chitosan is a mixture that does not give a sediment, trial results (figure 1) shows the ratio of extracts: chitosan which produces sediment is a ratio of 2 : 1 and 3 : 1, the full result can be seen in Table 2.

Tabel 2. The particles formula of chitosan coated corncob ethanol extract

Formula	The ration of chitosan and extract	Result
A. Levels of Corncob Ethanol Extract of 1 mg / mL		
F1A	1:3	Not precipitated
F2A	1:2	Not precipitated
F3A	1:1	Not precipitated
F4A	2:1	Precipitated
F5A	3:1	Precipitated
B. Levels of Corncob Ethanol Extract of 1,5 mg/mL		
F1B	1:3	Not precipitated
F2B	1:2	Not precipitated
F3B	1:1	Not precipitated
F4B	2:1	Precipitated

F5B	3:1	Precipitated
C. Levels of Corncob Ethanol Extract of 3 mg/mL		
F1C	1:3	Not precipitated
F2C	1:2	Not precipitated
F3C	1:1	Not precipitated
F4C	2:1	Precipitated
F5C	3:1	Precipitated
D. Levels of Corncob Ethanol Extract of 6 mg/mL		
F1D	1:3	Not precipitated
F2D	1:2	Not precipitated
F3D	1:1	Not precipitated
F4D	2:1	Precipitated
F5D	3:1	Precipitated
E. Levels of Corncob Ethanol Extract of 9 mg/mL		
F1E	1:3	Precipitated
F2E	1:2	Precipitated
F3E	1:1	Precipitated
F4E	2:1	Precipitated
F5E	3:1	Precipitated

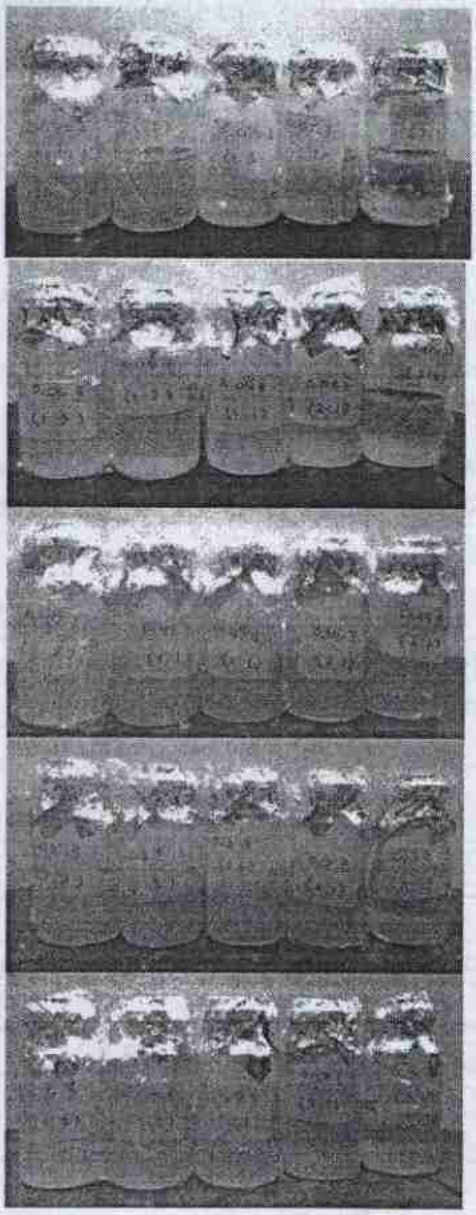

Figure 1. Particle of ethanol extract of chitosan coated corncob waste with various levels of extracts and various ratios of extract and chitosan. (a) solution level of corncob waste ethanol extract as much as 1.5 mg/mL, (b) solution level of corncob waste ethanol extract as much as 1 mg/mL, (c) solution level of corncob waste ethanol extract as much as 9 mg/mL, (d) solution level of corncob waste ethanol extract as much as 6 mg/mL, (e) solution level of corncob waste ethanol extract as much as 3 mg/mL.

Table 3. The result of Measurement test of chitosan coated corncob ethanol extract particles

Sample	Chitosan and extract ratio	The average particle size (nm)
Extract level 1 mg/mL	1:3	1300
Extract level 1 mg/mL	1:2	1198,2
Extract level 1 mg/mL	1:1	583,5
Extract level 6 mg/mL	1:3	1040,8
Extract level 6 mg/mL	1:2	572,6
Extract level 6 mg/mL	1:1	517,3

In particle size test(table 3.2), the researchers tried to measure the mixture of formula A and B in the ratio 1: 1, 1: 2 and 1: 3 since in the three comparisons the mixture does not experience precipitation. The resulting particles are still in microparticles (over 100 nm). This indicates the need for optimization of various extracts addition, chitosan and Na-TPP to produce nanoparticles and need to try to use other crosslinking such as pectin and alginate. From the result, the ethanol extract of corncob particles coated chitosan to be added in the cream is the ethanol extract of corncob with level of 6mg / mL with chitosan ratio 1:1.

Table 4. The Formula Optimization draft of Cream Preparation with Active Ingredient of Corncob Ethanol Extract microparticles based on design expert 9 along with the Response of Evaluation Test Result of Physical Cream Preparation.

Std	Run	Component 1 (Tween 80)	Component 2 (Span 80)	Response 1 (pH)	Renspon se 2 (Dispersive Power Test-cm^2)	Response 3 (Adhesiveness Test-second)	Response 4 (Viscosity -cps)	Response 5 (F)
6	3	8	8	7,26	3,325	2,50667	10005	0
1	6	1	15	7,01	3,625	0,89	11003	0
5	2	1	15	7,04	3,4	0,49	11197	0
4	5	11,5	4,5	7,28	2,825	1,82333	11370	0
2	4	4,5	11,5	7,29	3,475	0,746667	11485	0
3	7	15	1	7,09	3,525	1,19333	12107	0
7	1	15	1	7,06	3,225	1,00667	16487	0

The optimum formula consists of Tween 80 and Span 80 with a ratio 15 : 1. Optimum formula of cream preparation with active ingredents corncob ethanol extract nanoparticles then testing the total phenolic content (Figure 2).

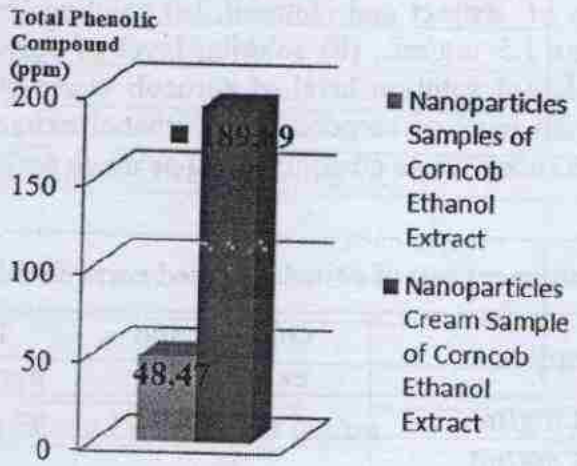

Figure 2. Comparison of Total phenolic Content of Corncob Ethanol Extract Nanoparticles And Cream Preparation With Nanoparticles Active Ingredients of Corncob Ethanol Extract

Conclusion

1. The smallest chitosan coated corncob extract Particles coated is produced by a mixture of ethanol extract of the corncob with levels of 6 mg / mL: chitosan (1: 1).
2. The optimum formula either cream base and cream preparation with active ingredients of corncob ethanol extract microparticles is the cream that contains tween 80 ratio and span 80 of 15:1.

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