



Research Article

FORMULATION OF BLACK HAIR DYES IN THE FORM OF STICKS FROM PAPAYA SEED EXTRACTS AND POWDER

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ABSTRACT

In general, Indonesian people assume that black hair is healthy, fertile, and beautiful hair, while hair that is not black, especially white, is considered unhealthy. Traditionally papaya seeds (*Carica papaya* L.) are used as a hair blackener, which is felt less practical because the material must be processed first by frying it without oil (roasting) and added with coconut oil, then applied to the hair. The purpose of this study was to formulate a black hair dye in the form of stick from papaya seed and its powder. The study began with the collection and determination of papaya sample. The papaya seed was milled and examined its characteristics on moisture content, ash content and phytochemical screening. Then the papaya seed extract was made by maceration with alcohol 70 %. Six formula black hair in sticks form was designed with various powder concentrations (25%, 35%, and 45%) and extracts (2.5%, 3.5%, and 4.5%) papaya seeds. Against the preparation, it was examined about the homogeneous test, consistency test, effectiveness test, and safety test. It was found that the water content of 4.66% w/w and ash content of 6.30% w/w were found in powder seed. Quinone was not found in the phytochemical screening. The six preparations made in a rod shape was good, stable, effective, and safe in its use. Statistically, it can be concluded that papaya seed extract can be formulated into hair blackening preparations that meet cosmetic requirements. The most effective and safe formula was a formula with 4.5% papaya seed extract.

Keywords: Blackened hair, papaya seed, *Carica papaya*, formulation, stick

INTRODUCTION

Hair is a valuable decoration for humans, especially for women hair is a mayor. Therefore, caring for your hair to thrive, healthy and healthy is very necessary. Hair is the most important part of mammals that has several functions such as protection or protection of the skin from mechanical disturbances and facilitates the regulation of temperature balance in the body or homeotherm¹⁻³. Most Indonesian people have black hair. Therefore, in general, people assume that black hair is healthy, fertile, and beautiful hair, while hair that is not especially white is considered unhealthy⁴. But this is not the case for elderly people, white hair or gray hair is a natural thing. But for those who are young, white hair, or graying is a problem. White or gray hair is common in humans. In fact, hair changes from black (original color) to white can occur not only in old age but also when young. These whitening changes occur due to pigment reduction in the hair follicles. Hair pigments are known as melanin when melanin production decreases, the hair will slowly turn white (gray hair)⁵. Graying of hair is usually progressive and permanent, but there are occasional reports of spontaneous repigmentation of gray hair⁶.

An overview of nutritional and phyto-therapeutic potential of papaya (*Carica papaya* Linn.) has been reported by Saeed *et.al*⁷ states that the seed could be used for gray hair treatment whereas Anjana *et.al*⁸ reviews on medical advantages and chemical constituents of *Carica papaya* Linn. Actually, Indonesian society traditionally uses a number of natural aspects of growing plants that can produce hair, or cover and prevent gray hair, including using tea water, green coconut water, straw, urang-aring (*Eclipta prostrata*), mangkokan (*Polyscias scutellaria*) leaves, hibiscus leaves, or with papaya seeds⁹. The use of papaya seeds as a hair

blackener is felt less practical because the material must be processed first by frying it without oil (roasting). Only then pounded until smooth, and when it will be used must be added with coconut oil, then applied to the hair and heads of the head while massaged shampooed, and rinsed thoroughly. This herb must be used every day until the hair or gray hair becomes blacker¹⁰.

This study reports on the formulation and testing of black hair dye formulas obtained from extract and the powder of papaya seeds in the stick form. Cosmetics preparation in the stick form is easy to use, practical and attractive. Hair coloring stick can be used directly on wet hair by rubbing it or on the hair of the forehead with a moistened brush first¹¹.

MATERIALS AND METHODS

Materials: The sample in the study was papaya seeds (*Carica papaya* L.) which had been smeared and dried. Drying papaya seeds by wind and not under direct sunlight.

Methods: Covered steps for preparing materials in the form of collecting papaya plants Bangkok varieties obtained from the Sukajadi area of Bandung, West Java and established in the toxicology laboratory, Biology Department, FMIPA, Universitas Padjadjaran. Papaya seeds were dried and made into powder. Fine powder (sieve No. 180, the nominal diameter of 0.131 mm wire) was made based on references from the Indonesian Ministry of Health¹² and Indonesian Materia Medika¹². The sample was determined by its characteristics in the form of water content, ash content based on Indonesian Pharmacopoeia IV¹³. Papaya seed powder was tested for its secondary metabolite content based on the Farnsworth method¹⁴. The Farnsworth method used included

detection of alkaloids by dissolving in 2N Hydrochloric acid and filtered. The filtrate was divided into four portions to achieve Dragendorff's test, Mayer's test, Wagner's test, and Hager's test; detection of flavonoids by treating with a few drops of sodium hydroxide solution. Formation of intense yellow color, which become colorless on addition of dilute acid, indicated the presence of flavonoids; detection of tannins by Gelatin test, Braymer's test, Ferric chloride-potassium ferricyanide test; detection of saponins by using Froth test: crude dry powder of extract is vigorously shaken with 2 mL of distilled water and was allowed to stand for 10 min. If stable froth appears, it indicated the presence of saponins; detection of quinones by adding about 5 mL of 10 % ammonium hydroxide solution was added to the filtrate, shaken and allowed to stand till the two layers are separated. The development of pink, to violet color in the ammonical phase indicated the presence of free quinones;

detection of triterpenoid by Libermann-Burchard test: The extract sample was dissolved in 2 mL of chloroform in a dry test tube. 10 drops of acetic anhydride and 2 drops of concentrated sulphuric acid were then added. If the solution became red, then blue and finally bluish-green in color, it indicated the presence of steroidal nucleus while the formation of purple or red color indicated the presence of a triterpenoidal nucleus. The Extraction was carried out by maceration with alcohol 70% according to Mustarichie *et.al* method¹⁵. The extract was dried with the aid of a rotary evaporator. Papaya seed extract and powder were then designed into 6 stick formulas. Making this formula referred to the Indonesian Cosmetics Formulary¹⁶ and the Indonesian Cosmetics Codex¹⁷. These formulas were then tested for stability test, homogeneity test, consistency test, and safety test. The results of this test were then concluded based on statistical methods¹⁸.

RESULTS

Table 1: Results of phytochemical screening of papaya seed powder

No	Secondary metabolites	Yield
1	Alkaloids	+
2	Flavonoids	+
3	Saponins	+
4	Tannins	+
5	Quinone	-
6	Triterpenoid	+

Notes: +: detected; -: not detected

Table 2: Basic formulations of hair dye sticks

Compounds	Amount of formula (%w/w)		
	FA	FB	FC
Triethanolamine	7.00	7.50	7.00
Glyceryl monostearate	7.50	6.00	8.00
Tragacanth	2.50	-	-
Stearic acid	13.50	15.00	15.00
Cera alba	47.78	43.78	49.08
Carnauba wax	13.50	-	-
Ozokerite	8.00	10.00	-
Coconut fatty acid	-	10.00	14.00
Methylparaben	0.20	0.20	0.20
Solid paraffin	-	7.50	6.00
Butyl Hydroxy Toluene	0.02	0.02	0.02

Table 3: Formula for black hair preparation in the form of sticks from papaya seed extract and powder

No	Compounds	Amount (% w/w)						
		F0	F1	F2	F3	F4	F5	F6
1	Triethanolamine	7.50	7.50	7.50	7.50	7.50	7.50	7.50
2	Glyceryl monostearate	8.00	8.00	8.00	8.00	8.00	8.00	8.00
3	Stearic acid	15.00	15.00	15.00	15.00	15.00	15.00	15.00
4	Cera alba	49.08	24.28	23.28	22.28	46.78	45.78	44.78
5	Diethylamide coconut fatty acids	14.00	14.00	14.00	14.00	14.00	14.00	14.00
6	Solid paraffin	6.00	6.00	6.00	6.00	6.00	6.00	6.00
7	Methylparaben	0.20	0.20	0.20	0.20	0.20	0.20	0.20
8	Butyl Hydroxy Toluene	0.02	0.02	0.02	0.02	0.02	0.02	0.02
9	Papaya seed powder	-	25.00	35.00	45.00	-	-	-
10	Papaya seed extract	-	-	-	-	2.50	3.50	4.50

Notes: F0 = Basic formula; F1 = Formula with papaya seed powder 25%; F2 = Formula with papaya seed powder 35%; F3 = Formula with papaya seed powder 45%; F4 = Formula with papaya seed extract 25%; F5 = Formula with papaya seed extract 35%; F6 = Formula with 45% papaya seed extract.

Table 4: Preparation Observation Results in Organoleptically for 2 months storage

Formula	Test	Storage time (days)							
		1	7	14	28	35	42	49	56
F1	Form	g	g	g	g	g	g	g	g
	Color	b	b	b	b	b	b	b	b
	Odor	sp	sp	sp	sp	sp	sp	sp	sp
	Homogeneity	h	h	h	h	h	h	h	h
F2	Form	g	g	g	g	g	g	g	g
	Color	b	b	b	b	b	b	b	b
	Odor	sp	sp	sp	sp	sp	sp	sp	sp
	Homogeneity	h	h	h	h	h	h	h	h
F3	Form	ng	ng	ng	ng	ng	ng	ng	ng
	Color	b	b	b	b	b	b	b	b
	Odor	sp	sp	sp	sp	sp	sp	sp	sp
	Homogeneity	h	h	h	h	h	h	h	h
F4	Form	g	g	g	g	g	g	g	g
	Color	pc	pc	pc	pc	pc	pc	pc	pc
	Odor	sp	sp	sp	sp	sp	sp	sp	sp
	Homogeneity	h	h	h	h	h	h	h	h
F5	Form	g	g	g	g	g	g	g	g
	Color	pc	pc	pc	pc	pc	pc	pc	pc
	Odor	sp	sp	sp	sp	sp	sp	sp	sp
	Homogeneity	h	h	h	h	h	h	h	h
F6	Form	g	g	g	g	g	g	g	g
	Color	pc	pc	pc	pc	pc	pc	pc	pc
	Odor	sp	sp	sp	sp	sp	sp	sp	sp
	Homogeneity	h	h	h	h	h	h	h	h

Notes: F1 Formula with 25% papaya seed powder, F2 Formula with concentration of 35% papaya seed powder, F3 Formula with 45% concentration of papaya seed powder, F4 Formula with 2.5% papaya seed extract, F5 Formula with 3.5% papaya seed extract, F6 Formula with 4.5% papaya seed extract
g: good; ng: deficient; b: black; pc: brownish white; sp: specific; h: homogen

Table 5: Results of the breaking point during storage time

Formula	Breakpoint during storage time on the day	7	14	21	28	35	42	49	56
	1								
FO	1501.3	1505.5	1507.9	1508.8	1511.7	1511.9	1512.3	1512.6	1513.7
F1	1225.1	1225.6	1226.9	1229.4	1231.2	1235.5	1240.0	1240.6	1240.9
1-2	1000.5	1000.9	1001.8	1004.6	1009.2	1018.4	1222.3	1228.7	1229.4
F3	982.7	982.9	983.4	986.3	988.9	990.8	995.2	997.7	998.2
F4	1486.3	1487.2	1489.9	1492.6	1493.3	1494.7	1495.4	1496.1	1496.8
F5	1475.8	1476.3	1477.1	1478.4	1479.0	1479.3	1480.7	1481.6	1482.5
F6	1453.1	1453.4	1454.1	1454.5	1454.9	1455.5	1455.8	1456.9	1457.2

Notes: Basic Formula FO, F1 Formula with 25% papaya seed powder, F2 Formula with a concentration of 35% papaya seed powder, F3 Formula with 45% concentration of papaya seed powder, F4 Formula with 2.5% papaya seed extract, F5 Formula with 3.5% papaya seed extract, F6 Formula with 4.5% papaya seed extract

Table 6: ANOVA calculation results

Variance source	dk	JK	KT	F	F _{table}	Notes:
						Hypothesis 0
Mean	1	329178181.9	32918181.9			
Treatment:						
A	6	7662489.7	1277081.617	362987.3308	2.17	Rejected
B	8	59385.1	7423.1375	2109.292454	2.01	Rejected
AB	48	237287.0	4943.479167	1405.094462	1.45	Rejected
Mistake	126	443.3	3.518253968			
Amount	189	14.8105				

DISCUSSION

Plant Determination Results: The results of the determination of all parts of the papaya plant used in the study were *Carica papaya* (Caricaceae). The determination was carried out at the Laboratory of Taxonomy, Department of Biology, Faculty of Mathematics and Natural Sciences, Universitas Padjadjaran, Bandung.

Powder characterization examination results: obtained water content of 4.66% w/w and ash content of 6.30% w/w. The water content obtained was in accordance with the literature that the

content of the water in the sample was not more than 10% ¹².

Phytochemical screening results: The results of phytochemical screening of papaya seed powder are shown in Table 1. Dada *et.al* ¹⁹ showed that there was no quinone in either papaya seed and peel in their phytochemical study on aqueous extract of *C. papaya* fruit's peel and seed. In Ghana, the study by Alorkpa *et.al* ²⁰ reported both ethanol and n-hexane extracts revealed the presence of alkaloids. Flavonoids, glycosides, and saponins were present in only the ethanol extract whereas tannins were present in the n-hexane extract. Tariq *et.al* ²¹, however, stated that the water extract of papaya seed only contained saponins and tannins. Most

likely, the different phytochemical screening results due to the origin of the analyzed papaya.

Extraction of Papaya Seeds: From 150 g of papaya seeds 15.01 g of thick brown extract was obtained with the taste and distinctive smell of papaya seeds, resulting in a yield of 10.07%.

Formulation of papaya seed extract and powder: First made 3 types of basic formulas (Table 2). The formulation was referred to lipstick formula²² and skin softening preparations²³. Then the three formulas compared their consistency for 7 days. On the last day, it was found the basic formula FC has the best consistency where there was no change in the shape and color of the stick made. The FC formula base was then used as the formula base. The base of the formula was then mixed with papaya powder and papaya seed extract as shown in Table 3. The principle of making this formula was Triethanolamine (TEA) and glyceryl monostearate heated on a water bath at 70 °C, then added with stearic acid and the temperature increased to 75 °C. Solid alba and paraffin were heated in a water bath at a temperature of 75-80 °C (mass 2). Mass 1 and mass 2 were mixed in hot conditions and other ingredients were added including papaya seed powder and extract with various variations of concentration, stirring until homogeneous. Pour into the mold at a temperature of 68-70 °C. The function of the chosen component was based on a standard formula of Indonesian Cosmetics Formulary⁵. TEA is used primarily as an emulsifier and surfactant. The formulation is mainly used as a pH adjusting agent. Other uses are as buffers, solvents, humectants, and polymer plasticizers. Used at concentrations of 2-4%. This substance can turn brown with exposure to air and light. Glyceryl stearate is a natural glyceryl ester from stearic acid (glycerine and stearic acid) which offers skin conditioning, moisturization and hydration due to glycerine components. Serves as a non-ionic opacifier, thickener, and stabilizer formulation, where it also infuses softer, smoother, feel for emulsions. Stearic acid is widely used for oral and topical formulations in pharmaceuticals. In topical formulations, stearic acid is used as an emulsion and solubilizing agent. The use of stearic acid in combination with TEA in cream preparations with a concentration of 1-20%. Coconut Fatty Acid Diethanolamide is a diethanolamine made by reacting a mixture of fatty acids from coconut oil with diethanolamine. Thick liquid and is used as a foaming agent in bath products such as shampoo and hand soap, and cosmetics as emulsifiers. The chemical formula of the individual component is $\text{CH}_3(\text{CH}_2)_n\text{C}(=\text{O})\text{N}(\text{CH}_2\text{CH}_2\text{OH})_2$, where n usually ranges from 8 to 18. The International Agency for Research on Cancer (IARC) lists coconut oil diethanolamine condensate (cocaide DEA) as an IARC Group 2B carcinogen, which identifies these chemicals that may be carcinogenic to humans. Paraffin is a mixture of hydrocarbons obtained from mineral oil. Consists of 2 forms, namely solid and liquid forms. Solid paraffin is used to harden the ointment because the melting point of the mixture will rise. Liquid paraffin consists of 2 types, namely the viscosity is dilute and viscous viscosity. Dilute viscosity is used for making vanishing cream, the viscosity is used for cold cream making. Parabens are used as preservatives in cosmetic and pharmaceutical products. Parabens help prevent mould and bacteria from arising, protect consumers, and maintain product quality. In chemical terms, parabens are esters of p-hydroxybenzoic acid. The types of parabens that are most often used in cosmetic products are methylparaben, Propylparaben, and butylparaben. Many care products contain parabens, such as shampoos, shaving gels, lubricants, pharmaceuticals, facial makeup, lotions, and toothpaste. BHT or butyl hydroxytoluene is a fat-soluble synthetic compound commonly used to preserve food and cosmetics to slow down the rate of autoxidation of ingredients in products that can cause changes in taste or color. As such, it is mainly used to prevent fats in food from becoming rancid - but also used in cosmetics, drugs, jet fuel, rubber, petroleum products, electrical transformer oils,

and embalming fluids. BHT (butylated hydroxytoluene) is an artificial laboratory chemical added to food as a preservative. Butylated hydroxytoluene (BHT), also known as dibutylhydroxytoluene, is a lipophilic organic compound, a phenol derivative chemical, which is useful for antioxidant properties. European and US regulations allow small amounts to be used as food additives. In addition to this usage, BHT is widely used to prevent oxidation in liquids (eg fuel, oil) and other materials where free radicals must be controlled^{24, 25}.

Formula physical stability test: This test included organoleptic test including form test, wrinkle, and odor, homogeneity test by splitting the preparation longitudinally and seen its homogeneity, and testing consistency with measured breaking points from the preparation. The test was carried out within 56 days of storage time. It was found that the form of each formula was stable during storage time, except in the formula F3 with 45% papaya seed powder where the dosage form was not good from the start because it was fragile and cracked. This was because the powder concentration was too large and the fat was too small so it could not be formed into a good stem preparation. During 56 days of storage time, it was found that all preparations were stable and no odor occurred (see Table 4). It showed that there was no oxidation reaction from the air to fat or oil as a carrier because in the formula an anti-oxidant butyl hydroxyl toluene was added and because the preservative added (methylparaben) was sufficient so that there was no oxidation against oil and fat could cause a rancid smell. Observations regarding homogeneity indicated that preparations made with various concentrations showed fairly good homogeneity during storage. This was evident by the absence of unwanted weak lumps.

The breaking point results are shown in Table 5. From Table 5 it could be explained that all formulas which showed the breaking point between 982-1514 grams, and all formulas experience the breaking point during storage time. The breaking point of the formula with papaya seed powder was smaller than the formula with papaya seed extract. This was due to the concentration of the powder added to the formula larger than the concentration of the extract, so powder formulas were more fragile. This meant that a number of sticks the one that was smeared was hard enough, but it was easily applied to the wet hair. From the statistical calculation using one-way ANOVA²⁶, it was found that $F_{\text{table}} (a = 0.05)$ smaller than F_{count} (see Table 6). This showed that there were differences real between the breakpoints of each formula made during the time of storage.

The results of observing the growth of fungi, the release of crystals or fluids during storage time: It was found that all the formulas made did not show any growth of fungi, crystalline crystals or liquid out for 2 months of storage. So it could be said that all formulas of various concentrations of papaya seed powder and extract made, were stable enough not to be overgrown with mushrooms, not crystals or brokers, during storage time. This showed that the concentration of preservatives and antioxidants added to the formula was sufficient.

Effectiveness and Security Testing Results: It was found when the formula was tried on 6 volunteers, a formula that was effective enough to give black to the hair was a formula with concentrated papaya seed extract 4.5% (F6). While other formulas were not effective. This was likely due to the form or type of preparation chosen which did not support the absorption or staining of the active substance in the hair or the added concentration of powder and papaya seed extract was less. However, if the concentration of powder or extract of papaya seeds was added more the possibility of the formula formed was not good (brittle or liquid). From the results of safety tests on 20 volunteers, it was found that the basic formula and the black hair preparation formula with various concentrations of papaya seed powder and extract did not

cause irritation, so it could be said that the black hair with various concentrations of papaya seed extract and the extract is safe to use. So far the black hair making an article from the papaya seeds extract and powder were new. Febriani²³ stated that a mixture of 5% concentration of avocado extract and 25% papaya latex powder could be made to stick for heel softener. It seemed research of the Febriani was the only formulation using papaya seed. Generally, the form of making stick form was used for deodorant preparation. Shahtalebi *et.al*²⁷, for example, reported their study on deodorant effects of an extract stick: Antibacterial activity and sensory evaluation of axillary deodorant. They concluded a single treatment with a stick deodorant containing dichloromethane sage extracted from 200, 400, or 600 µg / mL concentrations were effective in reducing the axillary level compared with the control, in healthy subjects. Imron *et.al*²⁸ formulated deodorant stick from *Aloe vera*. Zahara²⁹ study on the formulation of deodorant roll-on antiseptic preparation with Betel oil (*Piper betle* Linn.). Stick dosage forms were also numerous for lipstick formulations. Handayani *et.al*²⁹ used red dragon fruit extract as a blending agent on lipstick

CONCLUSION

Six formula black hair in sticks form was designed with various powder concentrations (25%, 35%, and 45%) and extracts (2.5%, 3.5%, and 4, 5%) papaya seeds. Against the preparation, it was examined about the homogeneous test, consistency test, effectiveness test, and safety test which was found as good, stable, effective, and safe in its use. Statistically, it can be concluded that papaya seed extract can be formulated into hair blackening preparations that meet cosmetic requirements. The most effective and safe formula was a formula with 4.5% papaya seed extract.

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