



ORIGINAL RESEARCH

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The valorization of neem leaves infused coconut oil at various concentrations for the production of natural liquid shampoo

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KEYWORDS

Coconut Oil
Infused Oil
Liquid Shampoo
Neem Leaves

ABSTRACT

The liquid shampoo is a product that is generally used to clean hair and scalp. The additional active ingredient used in the liquid shampoo is neem leaves which contain polyphenols that have antibacterial effects. Neem leaves extract was made by hot infusion method with coconut oil as a solvent. The purposes of this research were to find the best concentration of neem leaves infused oil for liquid shampoo material and compare the qualities of liquid shampoo with Indonesian National Standard (SNI) of Shampoo 06-2692-1992. The method of this research was a laboratory experiment with descriptive analysis. This research was conducted in five treatments. Concentration neem leaves infused oil for liquid shampoo materials were ratio between coconut oil and neem leaves in sample A = 720:0 (w/w); B = 720:22,5 (w/w); C = 720:30 (w/w); D = 720:45 (w/w); and E = 720:90 (w/w). The result showed that all of the shampoos met the SNI of Shampoo 06-2692-1992 according to organoleptic, moisture content, and pH. The best shampoo based on organoleptic observation was shampoo C (720:30 (w/w)) with a hedonic percentage of 40%, moisture content of 69.89%, pH 7.03, and specific gravity of 1.0223 g/g. The highest antibacterial activity against *S. aureus* was shampoo E (720:90 (w/w)) with an inhibition zone diameter was 9.5 mm. Shampoo with the highest antibacterial activity resulted from the highest addition of coconut oils infused Neem leaves. It proved that neem leaves were effective as an additive in making shampoo to boost its antimicrobial properties.

Introduction

A shampoo is typically a form of any ingredients used to wash and clean any dirt in hair and scalp (BSN, 1992). Four forms of shampoos are clear liquid shampoo, cream shampoo, gel shampoo, and dry shampoo (Tranggono and Latifah, 2007). The main ingredient of shampoo is a surfactant as a cleansing agent, and other ingredients are humectants, perfume, foam stabilizer, and pH adjuster. The most common surfactant used in shampoo is *Sodium Lauryl Sulfate* (SLS), known to have irritating potential, especially for dry and sensitive skin (Sasetyaningtyas, 2019).

There is a type of shampoo that does not use surfactant synthetic, such as SLS, which is called soap shampoo. This shampoo used fatty acid to be the main ingredient made with saponification

process (Tranggono & Latifah, 2007). Saponification is a process by which triglycerides are reacted with sodium or potassium hydroxide to produce soap (Alexander et al., 1964). The selection of oil as a raw material of shampoo is crucial for shampoo making process because it can affect the product qualities.

Coconut oil is a common material used in shampoo. Coconut oil has a high lauric acid concentration of 44 - 52% (Ketaren, 1986). Coconut oil known has good foam properties and natural antimicrobial. Coconut oil also has benefits for nourishing and softening hair. Therefore, coconut oil can be a suitable raw material for shampoo making.

Most of the shampoos today use SLS and also using chemical ingredients that have the potential for irritation. There are still few

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shampoos that have antibacterial properties, whereas bacteria is one of the reasons for scalp and hair problems. Bacteria infections such as *Staphylococcus aureus* can cause impetigo, cellulitis, abscess, and folliculitis in the scalp (Fatimah et al., 2016). Adding natural active ingredients can improve the functional properties of shampoo and make shampoo has antimicrobial activities and decrease the potential of irritation. Neem Leaves extract is one of the natural active ingredient sources without irritating potential. Neems are known to have many benefits for skin care, such as acne medication and face-hair masks. Neem Leaves extract has antibacterial properties against *Staphylococcus epidermidis* and *Propionibacterium acnes* (Sadiq and Azeem, 2017). It also contains flavonoid, saponin, and tannin as antibacterial (More et al., 2015). Neem also helps in strengthening hair quality and promotes the growth of hair. Due to its antibacterial, antifungal, and anti-inflammatory properties, neem is an excellent material to curb dandruff. Oil infusion with coconut oil is one method of extracting neem leaves, which involves soaking the leaves into the oil for 24 hours at high temperature to generate neem leaves infused oil in a slow cooker. According to Ellis (2014), infused oil is the oil that contains active compounds from herbal plants by the soaking method. In this method, coconut oil acts as a solvent for dissolving neem leaves compounds.

The shampoo has standard attributes which are determined by SNI Shampoo 06-2692-1992. These standards become a reference for making a good shampoo be a commercially viable product. Shampoo with neem leaves infused oil additional must fulfill the qualities standard of shampoo by SNI Shampoo 06-2692-1992. Based on the above description, research needs to be conducted to find the best concentration for adding neem leaves infused oil to liquid shampoo without compromising the product's quality.

Research Methods

Materials

The materials used in this research were coconut oil from the Nafisa brand and dried neem leaves (*Azadirachta indica*) from Asiatic's Cosmeceuticals & Herb.co. The chemical materials included Potassium hydroxide (KOH), glycerin (cosmetical grade), Propylene glycol,

distilled water, citric acid, lavender essential oil, Cocamide- DEA, concentrated HCl, concentrated sulfuric acid, FeCl₃, acetic anhydride, and nutrient agar (NA). The bacteria used for antibacterial activity analysis was *S. aureus*.

Methods

The method of this research was a laboratory experiment with descriptive analysis. There were five treatments namely the variation of concentration neem leaves infused oil for liquid shampoo materials were ratio between coconut oil and neem leaves in sample A = 720:0 (w/w); B = 720:22.5 (w/w); C = 720:30 (w/w); D = 720:45 (w/w); and E = 720:90 (w/w). The liquid shampoo formulations described in Table 1.

Neem Leaves Infused Oil

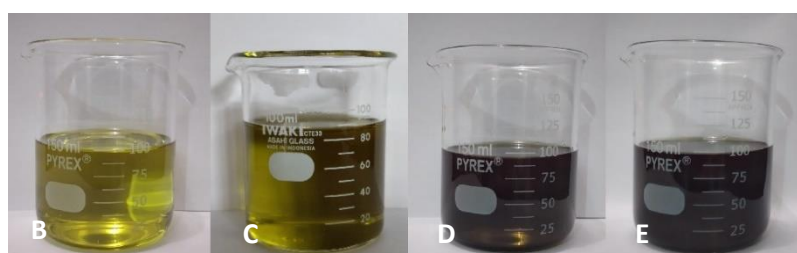
The dried neem leaves were ground into a fine powder using a grinder and sieved with 18 mesh to prepare the raw materials. The moisture content of neem leaves simplicia was then measured using a gravimetric method. Neem leaves infused oil made with heat infusion method with four treatments such as variation of concentration neem leaves infused oil for liquid shampoo materials were ratio between coconut oil and neem leaves in sample B = 720:22.5 (w/w); C = 720:30 (w/w); D = 720:45 (w/w); and E = 720:90 (w/w). The simplicia was soaked with coconut oil on a slow cooker for 24 hours with a warm heat indicator ($\pm 45^{\circ}\text{C}$). Neem leaves infused oil was then analyzed with screening phytochemicals method included flavonoid, tannin, saponin, and terpenoid tests.

Liquid Shampoo Making

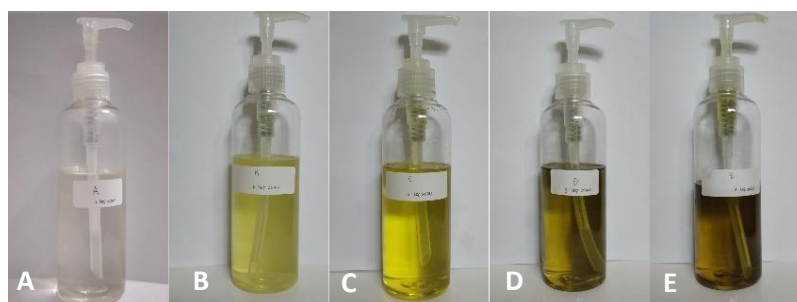
The heat method was used to make liquid shampoo, these include two key processes namely saponification and dilution Potassium hydroxide 36% solution was prepared by mixing potassium hydroxide flakes with distilled water. Saponification was the process of neem leaves infused oil and potassium hydroxide mixing on a slow cooker for ± 3 hours with a low heat indicator ($\pm 70^{\circ}\text{C}$) until produced pasta shampoo (traced). The process continues with diluted pasta shampoo with other ingredients such as glycerin, aquadest, propylene glycol, citric acid, cocamide DEA, and lavender oil used a slow cooker for an hour with a warm heat indicator ($\pm 45^{\circ}\text{C}$). Shampoo was placed in pump bottles and analyzed against the SNI of shampoo 06-2692-1992 standard.

Table 1. The formulation of liquid shampoos

Materials	Compositions (g)					Function
	A	B	C	D	E	
Neem leaves infused oil	75 (720:0 (w/w))	75 720:22.5 (w/w)	75 720:30 (w/w)	75 720:45 (w/w)	75 720:90 (w/w)	Oil with active ingredients
Potassium hydroxide 36% (w/v)	36	36	36	36	36	Alkaline
Glycerin	10.5	10.5	10.5	10.5	10.5	Humectant
Aquadest	141.7	141.7	141.7	141.7	141.7	Solvent
Propylene glycol	22.5	22.5	22.5	22.5	22.5	Humectant
Citric acid 25% (w/v)	5	5	5	5	5	pH adjuster
Coca – DEA	9	9	9	9	9	Foam stabilizer
Lavender oil	0.3	0.3	0.3	0.3	0.3	Fragrance

**Figure 1.** Neem Leaves Infused Oil

(B) 720:22.5 (w/w) (C) 720:30 (w/w) (D) 720:45 (w/w) (E) 720:90 (w/w)

**Figure 2.** Liquid Shampoo Appearances

(A) 720:0 (w/w) (B) 720:22.5 (w/w) (C) 720:30 (w/w) (D) 720:45 (w/w) (E) 720:90 (w/w)

Quality Analysis

The quality analysis was divided into physicochemical analysis, organoleptic, and antibacterial activity tests. The physicochemical analysis included moisture content, pH, specific gravity, viscosity, color, foam stability, and shampoo stability. The organoleptic test included color, odor, texture, foam, and usage impression observed by 30 panelists. The criteria of panelists such that panelists must be healthy (not influenza or sick), panelist not using perfume before the test, and panelist not using scent soap before the test. The panelists rated each shampoo subjectively. Panelists rated each shampoo with hedonic scale from 1 to 5, where 1 = very dislike, 2 = dislike, 3 = neither like nor dislike, 4 = like,

and 5 = very like. Moreover, the panelist gave rank for each shampoo from 1st to 5th.

An antibacterial activity test used the disk diffusion method and the bacteria used on the test was *S.aureus*. The antibacterial activities were evaluated using the agar diffusion test with extract at five different concentrations A = 720:0 (w/w); B = 720:22.5 (w/w); C = 720:30 (w/w); D = 720:45 (w/w); E = 720:90 (w/w).

Results and Discussion

The Yield of Neem Leaves Infused Oil and Liquid Shampoo

In the process of making neem leaves infused oil, the slow cooker's average temperature was

52.45°C \pm 0.91. Neem leaves used for infused oil had a moisture content of 8.64 \pm 0.051% that was measured by the gravimetric method. In the making shampoo process, the average temperature at saponification process was 72.24°C \pm 0.622 and the dilution process was 50.20°C \pm 1.41. The results of neem leaves infused oil and liquid shampoo are shown in Figures 1 and 2.

Figure 3 showed that a higher yield percentage resulted from the high concentration of neem leaves infused oil. The appearance of neem leaves infused oil and liquid shampoo showed that the higher concentration treatment has resulted in

a deeper color. It was caused by the high concentration containing more active compounds. The result of phytochemical screening showed that infused oil was positive for tannin, but flavonoid, saponin, and terpenoid were not detected. The tannins could be carried by non-polar solvents like oil up to 20% (Singh et al., 2011). The phytochemical component of neem leaves extract have been established in previous studies, these include saponin, tannin, alkaloid, phenols (Biswas et al., 2002).

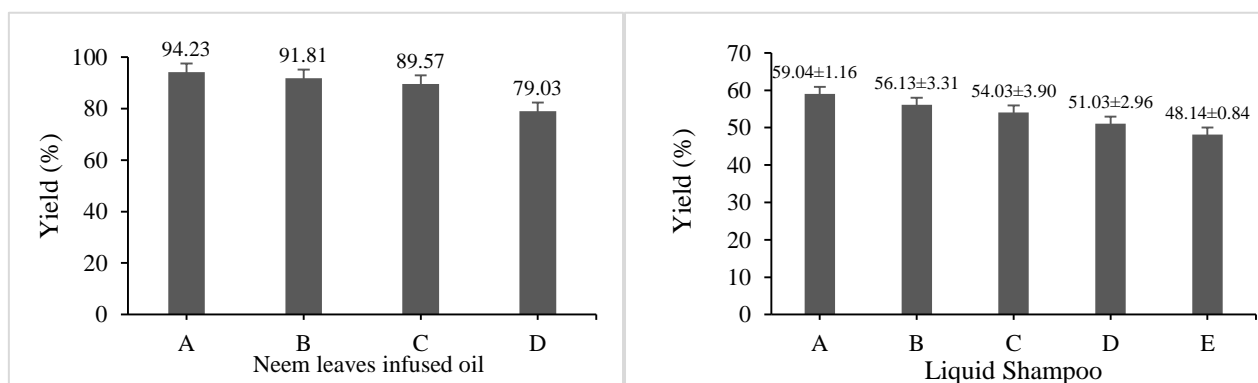


Figure 3. Yield of neem leaves infused oil and liquid shampoo

Table 2. Physicochemicals Profiles of Liquid Shampoo

Sample	Parameters					SNI of Shampoo 06-2692-1992
	A	B	C	D	E	
Moisture Content (%)	70.22	71.00	69.89	71.56	70.66	Max. 95%
pH	7.37	7.20	7.03	6.96	6.86	5 – 9
Specific Gravity	1.0197	1.0215	1.0223	1.0232	1.0245	Min. 1.02
Viscosity (centipoise)	310	280	400	420	435	-
Color	Clear	Yellow	Yellow	Yellow	Yellow	-
Foam Stability (%)	82.75	80.95	82.13	83.22	83.51	-
Shampoo stability	Stable	Stable	Stable	Stable	Stable	-

A = 720:0 (w/w); B = 720:22.5 (w/w); C = 720:30 (w/w); D = 720:45 (w/w); E = 720:90 (w/w)

Physicochemical of Liquid Shampoos

The physicochemical analysis included moisture content using the gravimetric method, pH used pH meter, specific gravity used pycnometer, viscosity used viscometer, color, foam stability with cylinder shake method, and shampoo stability during 14 days of storage. The results of the physicochemical analysis was listed in Table 2.

Based on SNI Standard, shampoo should have max. 95% for moisture content, 5 - 9 for pH, and min. 1.02 for specific gravity. The moisture content result of each shampoo in Table 2 showed fluctuation in value due to the difference in evaporation at the saponification process. The

differences in the time duration of stirred process at saponification made the material evaporation of each shampoo was different. According to moisture content results showed that all produced shampoos were qualified based on SNI Shampoo 06-2692-1992. The data in Table 2 described that pH decrease at the higher concentration of neem leaves infused oil. Because the tannins in the neem leaves infused oil were acidic, the pH dropped. 1% solution of tannin has a pH of 4.56 making it classified as weak acids (Sampepana and Rosarnah, 2010). Specific gravity results in Table 2 showed that all shampoo qualified by SNI shampoo except shampoo A. Specific gravity of

shampoo A was 1,0197 g/g while the standard was min. 1.02 g/g.

Specific gravity increase at the higher neem leaves infused oil concentration. Specific gravity depends on the amount of active compounds on the materials, which means a higher concentration would increase specific gravity (Sheikh and Bath, 2012). Specific gravity was directly proportional

to viscosity. In Table 2, it is shown that the viscosity value increases with increasing specific gravity except for shampoo B. It is caused by a higher moisture content value in shampoo B if compared with shampoo A. It also affects the foam stability, where the foam stability in shampoo B has the lowest value than others.

Table 3. Organoleptic test of liquid shampoo

Parameters	Average Score				
	A	B	C	D	E
Color	4	3	5	4	3
Odor	3	3	4	4	4
Texture	3	3	4	4	4
Foam	4	4	4	5	4
Usage Impression	4	4	5	4	4

Table 4. Percentage of panelist rank preference

Parameters	Shampoo				
	A	B	C	D	E
1 st Rank	13	17	40	20	13
2 nd Rank	13	17	27	33	23
3 rd Rank	23	17	17	17	27
4 th Rank	20	27	10	17	23
5 th Rank	30	23	7	13	13

Based on color results and shampoo stability for 14 days, shampoo A was clear where other shampoos were yellows determined by chromameter. Furthermore, the shampoo stability in 14 days was observed by stabilities of color, texture, and odor. The results showed each shampoo was stable and not changed based on color, texture, and odor.

Organoleptic of Liquid Shampoo

Organoleptic tests observed color, texture, odor, foam, usage impression subjectively by 30 panelists. The result of an organoleptic test in Table 3 and Table 4 showed the best shampoo was shampoo C by a percentage of rank preference by panelists was 40% for 1st rank. Shampoo C has a high score than others, where the color and usage impression has an average score of 5, and other parameters (odor, texture, and foam) of 4. No shampoo has a low score from all parameters, which indicates that the shampoos qualified the consumer's requirements.

Antibacterial Activity

The antibacterial activity test used the disk diffusion method with *S.aureus* as a tested bacteria. *S. aureus* was one of the bacteria on the scalp and caused scalp health problems (Fatimah et al.,

2016). The result of antibacterial activity in liquid shampoo showed in Figures 4 and 5.

The result showed a concentration of neem leaves infused oil directly proportional to inhibition zone diameters. Antibacterial activity on shampoo due to neem leaves infused oil that contains tannin, high concentration treatment made antibacterial activity was getting stronger because more tannins in the neem leave infused oil. Tannins also can inactivate bacterial enzymes and disrupt the passage of proteins in the cell layer (Ngajow et al., 2013). Tannins in neem leaves extract are known to have antibacterial properties and can inhibit *Salmonella typhi* bacteria (Ruwandha et al., 2021). All of them were classified into moderate antibacterial properties with an inhibition zone between 5 – 10 mm (Davis and Stourt, 1971).

Conclusions

The shampoos with all concentrations of neem leaves infused oil met the SNI Shampoo requirements (moisture content, pH, and specific gravity), and organoleptic parameters). The best treatment was shampoo C (720:30 w/w) with a percentage of rank preference of 40% in the organoleptic test, moisture content of 69.89%; pH of 7.02; specific gravity was 1.0223 g/g; also inhibition zone diameter of 8 mm. Shampoo with the highest antibacterial activity resulted from the

addition of infused oil with the highest concentration, namely 720:90 (w/w) in treatment E with an inhibition zone diameter of 9.5 mm.

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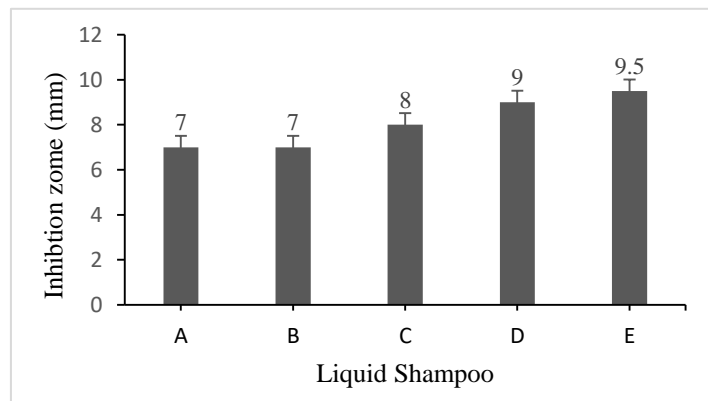


Figure 4. Inhibition Zone of *S.aureus*

(A) = 720:0 (w/w); (B) = 720:22,5 (w/w); (C) = 720:30 (w/w); (D) = 720:45 (w/w); (E) = 720:90 (w/w)

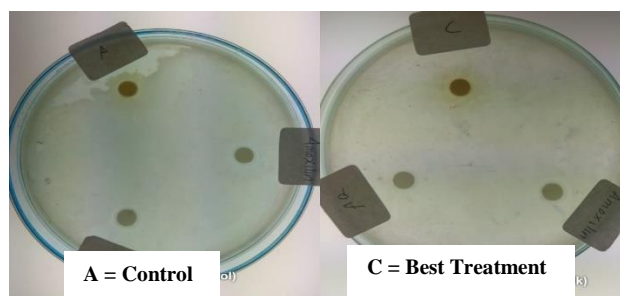


Figure 5. Inhibition Zone of (A) Control and (C) Best Treatment

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