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Effect Of Propylene Glycol On The Physical Preparation Of Katuk Leaf Ethanol Extract Herbal Syrup

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ABSTRACT
Katuk has been used by the Indonesian citizen as a breast milk enhancer for breastfeeding mothers. People consume katuk in the form of decoction, herbal tea, or capsules. These preparations has a strong katuk taste and aroma. Preparation of ethanol extract of katuk leaves in herbal syrup has not been developed yet. The advantage of syrup is that it can cover unpleasant odors and aromas. Propylene glycol is an additional ingredients in the syrup which functions as a cosolvent which can increase solubility. The aim of this research was to determine the effect of propylene glycol on the physical preparation of katuk leaf ethanol extract herbal syrup. The herbal syrup formula for ethanol extract of katuk leaves contains katuk leaf extract, sucrose, anhydrous citric acid, methylparaben, propylparaben, glycerin, peppermint oil, distilled water, and propylene glycol with varying concentrations between 5-20%. The results of the research showed that extract yield of 10.6%. Katuk leaves extract ethanol contain flavonoids, phenolics, alkaloids, saponins, and tannins. The results of the physical preparation test of katuk leaf ethanol extract herbal syrup showed liquid form, green color, katuk leaf aroma, pH of around 6.16-6.20, specific gravity of around 1.1673-1.2523 g/mL, viscosity of around of 5.53-16.00 cPs, clarity, homogeneous, and displaced volume is 100 mL. The conclusion obtained is that the difference in propylene glycol composition does not affect the shape, color, aroma, pH, clarity, homogeneity, and displaced volume test. The greater the propylene glycol composition, the smaller the specific gravity and viscosity.

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INTRODUCTION

Katuk (*Sauropus androgynus* (L.) Merr.) is a plant that is easy to grow in Indonesia and is widely known by people in West Asian and Southeast Asian countries and has been used by the Indonesia citizen because it has various pharmacological activities, one of which is facilitating breast milk in nursing mothers (Ramadheni et al., 2018). Katuk plants have a height of around 50 cm to 3.5 m, their stems grow upright and woody, and the leaves are small and green (Ahmad & Nuraeni, 2015). Katuk leaves are rich in flavonoids, saponins, tannins, alkaloids, minerals, and various vitamins such as vitamin A, vitamin B1, and vitamin C (Ahmad & Nuraeni, 2015). Active compounds in katuk leaves include glycosides,

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saponins, tannins, flavonoids, steroids, alkaloids, carbohydrates, and proteins, which have antioxidant, anti-inflammatory, antimicrobial properties, antidiabetic, antiobesity, and lactation inducing (Majid & Muchtaridi, 2018). To meet the adequacy of breast milk for breastfeeding mothers, one way that has been found is to consume katuk leaves. Katuk leaves contain sterols and alkaloids which can increase breast milk production. The sterol content can increase glucose metabolism for lactose synthesis so that breast milk production increases. It also contains polyphenols and steroids which play a role in the prolactin reflex or stimulate the hormone oxytocin to stimulate the release and flow of breast milk. 100 grams of katuk leaves contain 4.8 grams of protein, 2 grams of fat, 11 grams of carbohydrates, 2.2 grams of minerals, 24 mg of calcium , 83 mg phosphorus, 2.7 mg iron, 31.11 µg vitamin D, 0.10 mg vitamin B6 200 mg vitamin C, 72 calories, and 70 grams of water (Ahmad & Nuraeni, 2015). Katuk leaf ethanol extract contains alkaloids, flavonoids, saponins, quinones, tannins, triterpenoids and steroids and has antifungal and antibacterial activity (Japa et al., 2019; Ramadheni et al., 2018).

Syrup is a liquid preparation in the form of a solution containing sucrose, unless stated otherwise, the sucrose content, $C_{12}H_{22}O_{11}$ is not less than 64% and not more than 66.0% (Departemen Kesehatan Republik Indonesia, 2020). Syrup is an oral solution that contains high levels of sucrose or other sugars which is 64-66% (Departemen Kesehatan Republik Indonesia, 2020). Syrup consists of active substances, solvents, sweeteners, stabilizers, preservatives, thickeners, colorings, fragrances, flavors and isotonics. Active substances are the main substances/substances that are efficacious in syrup preparations. Solvents are liquids that can dissolve active substances or are usually referred to as carrier substances. Examples of solvents are water, glycerol, propylene glycol, ethanol, ether. Sweeteners are additional substances in a syrup, sweeteners are added to give the syrup a sweet taste. Stabilizers are intended to keep the syrup in a stable state. Examples of stabilizers are antioxidants, buffers, complexants. Preservatives are added to syrup preparations so that the syrup lasts longer and can be used repeatedly.

The aim of this research was to determine the effect of differences in propylene glycol composition on the test results of physical preparations of katuk leaf ethanol extract herbal syrup which included organoleptic, pH, specific gravity, viscosity tests, clarity, homogeneity, and displaced volume.

METHODS

Equipment and Material

The tools used are analytical scales, blenders, beakers, stirring rods, Buchner funnels, rotary evaporators, water baths, test tube racks, test tubes, bunsens, mortars, stampers, beakers and water baths. The materials used are katuk leaves, 96% ethanol, filter paper, sulfuric acid, hydrochloric acid, Mg powder, sodium hydroxide, 5% $FeCl_3$, chloroform, 10% HCl, Meyer's reagent, 70% ethanol, anhydrous citric acid, methyl and propyl parabens, glycerin, propylene glycol, sucrose, peppermint oil, and distilled water.

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Production of Katuk Leaf Ethanol Extract

The extraction method used is based on the modification of extraction method carried out by Nurdianti & Tuslinah (2017). Prepare 500 grams of katuk leaves. Katuk leaves are cut into small pieces with scissors and washed until clean. Next, the katuk leaves are dried by heating them at 50 °C in an oven for about 16 hours. Once dry, blend it until it forms a powder and then sift it with a 40 mesh sieve. Carefully weigh 50 grams of simplicia powder then put it in a 500 mL beaker. Add 450 mL of 96% ethanol into the beaker. Stir the marinade until homogeneous then leave for 24 hours. After letting it sit for 24 hours, filter the extract using a Buchner funnel, then the filtrate is taken to thicken with a rotary evaporator at a temperature of 60°C until the extract is thick but can still be poured from the rotary evaporator. The 96% ethanol residue is then evaporated using a water bath. The method is to put the thick extract into a porcelain cup, then stir the extract with a stir stick until thick.

Phytochemical Screening

Phytochemical screening was carried out based on the method carried out by (Pamungkas et al., 2016) :

a. Identification of flavonoid compounds
   Put 2 mL of the extract solution into a test tube. Do this 3x. In tube 1, add 5 mL of concentrated H_2SO_4, tube 2 add 5 mL of concentrated HCl and add Mg powder, tube 3 add 5 mL of NaOH. The presence of flavonoids is indicated by the appearance of red, yellow or orange colors.
b. Identification of phenolic compounds
   Put 1 mL of the extract solution into a test tube then add 2 drops of 5% $FeCl_3$ solution. A positive reaction is indicated by the formation of a green or blue-green color.
c. Identification of alkaloid compounds
   Put 100 mg of thick extract into an Erlenmeyer flask then add 20 mL chloroform, filter and add 10 mL of 10% HCl to the filtrate. The extraction results will form two layers, then take 5 mL of the HCl layer and put it in a test tube. And added Meyer's reagent. If a white precipitate forms, it is positive for alkaloids.
d. Identification of saponin compounds
   Put 100 mg of thick extract into a test tube, then add 10 mL of distilled water and heat over a Bunsen. After that, 5 mL of the water phase was taken, shaken vigorously. If there is foam formed, then add 1 drop of concentrated HCl while there is still foam then this shows that it positively contains saponin.
e. Identification of tannin compounds
   A sample of 1 mL of thick ethanol extract was put into a test tube, then 2 mL of 70% ethanol was added, then stirred, 3 drops of $FeCl_3$ were added to the extract. If there is a change in color from blue-black, green or blue-green and sediment then it indicates tannins in general.

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Production of Katuk Leaf Ethanol Extract Herbal Syrup

The formula and method for making katuk leaf ethanol extract herbal syrup is based on the modification of formula and working method carried out by (Artania et al., 2020). Simplex syrup is made first in a separate container. Mixture A contain the thick extract of katuk leaves is mixed with propylene glycol while being crushed in a mortar. In a separate mortar, grind anhydrous citric acid, methyl paraben, and propyl paraben until homogeneous. Mixture B contain glycerin and peppermint oil then grind again until homogeneous. Mixture A and mixture B are then put into a glass beaker and placed on a water bath then stirred until homogeneous. Simplex syrup is added to the mixture of ingredients and then stirred until a homogeneous simplex syrup is obtained. The mixture was put into a 100 mL glass bottle which had previously been weighed and then added with distilled water to the mark. Shake until homogeneous.

Table 1. Katuk Leaf Ethanol Extract Herbal Syrup Formula

No	Material	Formula 1	Formula 2	Formula 3	Formula 4	Formula 5
1	Katuk leaf ethanol extract	1,33 %	1,33 %	1,33 %	1,33 %	1,33 %
2	Sucrose	67%	67%	67%	67%	67%
3	Anhydrous citric acid	2%	2%	2%	2%	2%
4	Methyl paraben	0,18%	0,18%	0,18%	0,18%	0,18%
5	Propyl paraben	0,02%	0,02%	0,02%	0,02%	0,02%
6	Gliserin	3%	3%	3%	3%	3%
7	Propylene glycol	5%	10%	15%	20%	25%
8	Peppermint oil	0,05%	0,05%	0,05%	0,05%	0,05%
9	Aquadest	ad 100 mL	ad 100 mL	ad 100 mL	ad 100 mL	ad 100 mL

Physical Preparation Test of Katuk Leaf Ethanol Extract Herbal Syrup

a. Organoleptic test
   Organoleptic tests on syrup preparations include taste, smell and color as an indicator of subjective physical properties (Sayuti & Winarso, 2014)
b. Test pH
   Dip the pH meter probe into the syrup solution, record the pH.
c. Spesific gravity test
   The specific gravity test is carried out using a clean and dry pycnometer. The syrup solution is put into the pycnometer. Adjust the temperature of the charged pycnometer to 20°C, remove excess liquid and weigh. If the monograph indicates temperature other than 20°C, the charged pycnometer must be adjusted until the desired temperature is reached before weighing. Subtract the weight of the empty pycnometer from the weight of the full pycnometer. The specific gravity of a substance is the result obtained by dividing the weight of the substance by its weight water in the pycnometer (Fitriana et al., 2022)

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d. Viscosity test
First, 200 mL of the sample is placed in a beaker glass, then the spindle is installed and the revolver is rotated until the spindle is submerged at the spindle boundary line with the spindle head positioned in the middle. The speed is set at 100 rpm. The viscosity data results use cPoise (cP) units.

e. Clarity test
The clarity test is carried out visually by observing the preparation. Preparation test results the syrup should be clear, and free from impurities (Fickri, 2019)

f. Homogeneity test
The homogeneity test was carried out on the finished syrup which was given until 50 ml in a container. The container is shaken and then observed to see whether it is homogeneous. The test is repeated three times. A good syrup must be stable, homogeneous, free from turbidity and free from contamination and microbial growth (Hidayati et al., 2020)

g. Displaced volume test
The displaced volume test is designed to ensure that oral solutions and syrups packaged in multidose containers, with a labeled volume of no more than100 ml. Available in liquid or fluid dosage forms. medium. Indicated volume, moment removed from the original container, giving the volume stated on the preparation label (Helni, 2013).

RESULTS AND DISCUSSION

The extraction method used to extract phytochemical compounds in katuk leaves is maceration. The maceration extraction method was chosen because the method and equipment are simple, it does not use heating so it can prevent the decomposition of the active substances contained in the sample due to the influence of temperature and compounds that cannot withstand heating (Sa'adah & Nurhasnawati, 2015). The maceration method is very beneficial in isolating natural compounds because by immersing the sample, the cell walls are broken down due to the pressure difference inside and outside the cell so that secondary metabolites in the cytoplasm dissolve in organic solvents and compound extraction will be perfect (Lenny, 2006). Maceration was carried out using 96% ethanol solvent. Ethanol is used as a solvent because it is universal, polar and easy to obtain. 96% ethanol was chosen because it is selective, non-toxic, has good absorption and high filtering ability so that it can filter out non-polar, semi-polar and polar compounds compared to 70% ethanol which is more polar and therefore extracts more polar compounds.. The 96% ethanol solvent penetrates more easily into the cell walls of the sample than the ethanol solvent with a lower concentration, resulting in a concentrated extract. The maceration results are then evaporated using an oven at a temperature of 50°C to obtain an ethanol extract. The yield of katuk leaf ethanol extract produced was 10.60%. The extract obtained is dark green in color with a distinctive katuk leaf aroma. The form of the extract obtained is the same as the form of the extract obtained by Susanti et al. (2014) namely dark green.

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Table 2. Phytochemical Screening Result of Katuk Leaf Ethanol Extract

Compound	Reference	Result	Conclusion
Flavonoid	Red, yellow, or orange	Orange	Positive
Phenolic	Green to blue	Blue	Positive
Alkaloid	White precipitate	White precipitate	Positive
Saponin	Foam	Foam	Positive
Tannin	Blue to black, green, blue	Black	Positive

To determine the phytochemical content in the ethanol extract of katuk leaves, a phytochemical screening was carried out. Table 2 shows that katuk leaves contain flavonoids, phenolics, alkaloids, saponins and tannins. The results of the phytochemical screening test for the ethanol extract of katuk leaves obtained from this study were the same as the test results carried out by Susanti et al. (2014) which also show positive result for flavonoids, phenolics, alkaloids, saponins and tannins.

Table 3. Physical Preparation Test Results

Parameter	Formula 1	Formula 2	Formula 3	Formula 4	Formula 5
Form	Liquid	Liquid	Liquid	Liquid	Liquid
Colour	Green	Green	Green	Green	Green
Odor	Katuk leave	Katuk leave	Katuk leave	Katuk leave	Katuk leave
pH	6.18	6.20	6.19	6.16	6.18
Specific gravity	1.2523	1.2512	1.2506	1.2018	1.1673
Viscosity	16.00	13.99	9.17	5.93	5.53
Clarity	Clarity	Clarity	Clarity	Clarity	Clarity
Homogeneity	Homogenous	Homogenous	Homogenous	Homogenous	Homogenous
Displaced Volume Test	100 mL	100 mL	100 mL	100 mL	100 mL

The physical preparation tests carried out are organoleptic, pH, specific gravity, viscosity, clarity, homogeneity, and displaced volume test. The organoleptic test results of the ethanol extract of katuk leaf herbal syrup showed that it was liquid, green in color, and had the aroma of katuk leaves. The difference in propylene glycol composition did not affect the organoleptic test results.

pH is acidity, which is used to express the acidity of a solution (Nuzzaibah & Ermawati, 2023). The pH test aims to determine the pH of the product which is related to the solubility of the substance active stability (Nuzzaibah & Ermawati, 2023). pH testing is one of important parameter because the stabil pH value of the solution shows that the distribution process of basic materials in even preparation (Nuzzaibah & Ermawati, 2023). pH measurements are carried out using a pH meter. The pH range obtained between formula 1 to formula 5 is 6.16 to 6.20, so it falls within the acceptable syrup pH range requirements between 4-7 (Departemen Kesehatan Republik Indonesia, 2020). Differences in propylene glycol composition do not affect pH katuk leaves ethanol extract syrup.

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The weight uniformity test is carried out to control the specific gravity of the syrup preparation (Nuzzaibah & Ermawati, 2023). Specific gravity assessment is made with a pycnometer, the specific gravity requirements for syrup preparations are ≥ 1.2 g/mL (Nuzzaibah & Ermawati, 2023). The specific gravity test result range obtained between formula 1 to formula 5 is 1.2523 g/mL to 1.1673 g/mL. Only formula 1 until formula 4 meet the good specific gravity specifications, namely more than 1.2 g/mL. The greater the propylene glycol composition, the smaller the specific gravity.

The viscosity test is important to carry out on syrup preparations because it is one of the quality control parameters in production activities (Nuzzaibah & Ermawati, 2023). The quality of the syrup preparation becomes not better when the viscosity is less than the standard value ultimately making the preparation very easy to pour when the viscosity exceeds the standard value then the syrup will be too thick to pour, allowing the presence of an active substance left in the preparation container (Nuzzaibah & Ermawati, 2023). In oral liquid preparations, the viscosity needs to be increased for easy consumption and improve pouring. The requirements for good viscosity are 10-30 cps (Nuzzaibah & Ermawati, 2023). Viscosity tests are carried out for ensure the viscosity of this test preparation done using tools Brookfield viscometer. The viscosity test result range obtained between formula 1 to formula 5 is 16.00 cPs to 5.53 cPs. Only formula 1 and formula 2 meet the good viscosity specifications, namely between 10-30 cps. The results can be concluded by adding the concentration of propylene glycol, the higher the concentration, the lower the viscosity value.

The clarity test is carried out visually by observing the preparation. Preparation test results of the syrup should be clear and free from impurities (Nuzzaibah & Ermawati, 2023). The results show that the syrup preparation for each particle formula is distributed uniformly equally because not all formulations have coarse grains or flying particles. Therefore, it can be concluded that the five resulting formulas have clarity the good one.

The homogeneity of syrup preparations is closely related to the solubility of various substances contained in the syrup with the solvent used (Nuzzaibah & Ermawati, 2023). In this case point, the biggest pressure is the solubility of lime leaf extract towards the main solvent syrup is water (Nuzzaibah & Ermawati, 2023). This test was carried out because of important requirements in the solution preparation that is, homogeneous. Observation results on the five syrup formulas shows that the resulting syrup preparation is a homogeneous colored liquid dark green without any sediment found after making syrup.

The displaced volume test is carried out to determine how easy the solution is poured, the more difficult the calculation, the thicker the solution, whereas the easier it is to pour, the lower the viscosity or the more fluid it will be lots (Nuzzaibah & Ermawati, 2023). The more liquid, the easier it is to pour. Volume requirements transferred to the syrup preparation is less than 100%, not less than 95% the volume is indicated on the label (Nuzzaibah & Ermawati, 2023). Based on the data it can be concluded that the all displaced volume of syrup preparation still meets the requirements.

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CONCLUSION

The extract yield of katuk leaves is 10.6%. Based on phytochemical screening result, katuk leaves extract ethanol contain flavonoids, phenolics, alkaloids, saponins, and tannins. The results of the physical preparation test of katuk leaf ethanol extract herbal syrup showed liquid form, green color, katuk leaf aroma, pH of around 6.16-6.20, specific gravity of around 1.1673-1.2523 g/mL, viscosity of around of 5.53-16.00 cPs, clarity, homogeny, and volume displaced test is 100 mL. The conclusion obtained is that the difference in propylene glycol composition does not affect the shape, color, aroma, pH, clarity, homogeneity, and volume displaced test. The greater the propylene glycol composition, the smaller the specific gravity and viscosity.

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