A STUDY ON ULTRASONIC ASSISTED EXTRACTION AND FORMULATE NATURAL HAIR SHAMPOO FROM SAPINDUS EMARGINATUS

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ABSTRACT

Hair shampoo is a personal care product that can removes dirt, skin particle, and sebum from hair skin. Mostly of hair shampoo consist of dangerous ingredient such as Sodium Laureth Sulfate which it produces skin and hair damages, including cracking and severe inflammation of tissue. Thus, Sapindus emarginatus will be used as main ingredient for a new formulation of hair shampoo. This herb material contains an active compound known as saponin which can replace the dangerous compound in hair shampoo. The objective of this research is to extract saponin from Sapindus emarginatus using ultrasound assisted extraction in order to formulate natural hair shampoo. The yields of saponin was measured by HPLC by study the effect of different size particle, sonication time and temperature. Based on result, the maximum amount of saponin extracted obtained at size particle 315 µm, sonication time at 60 minutes and temperature at 60°C. The herbal extract was mix with other ingredients such as Methyl paraben and EDTA to form of a new formulation. The shampoo was tested using pH test. The extraction rate constant, k of saponin decreased with increasing temperature and sonication time, and the k values between range 0.1091 to 0.430. Thus, the saponin with higher concentration is used as main ingredient in order to formulate natural hair shampoo.
ABSTRAK

Syampu rambut merupakan produk penjagaan diri yang boleh menghilangkan kotoran, kotoran pada zarah kulit dan sebum daripada kulit rambut. Kebanyakannya syampu rambut terdiri daripada bahan berbahaya seperti Sodium Laureth Sulfate yang menghasilkan kerosakan kulit dan rambut termasuk keradangan retak dan tisu yang teruk. Oleh itu, sapindus emarginatus akan digunakan sebagai bahan utama untuk formulasi syampu rambut yang baru. Bahan herba ini mengandungi sejenis bahan aktif yang dikenali sebagai saponin yang boleh menggantikan komponen berbahaya di dalam syampu rambut. Objective bagi kajian ini adalah untuk mengeluarkan saponin daripada Sapindus emarginatus menggunakan ultrasonic dalam membuat formula shampoo asli. Untuk kaedah, dalam usaha untuk mendapatkan komponen bioaktif ini, sapindus emarginatus perlu dalam bentuk serbu. Apabila masa semakin meningkat, terdapat perubahan pada suhu. Semakin tinggi suhu, semakin maksimum hasil yang diperolehi bagi masa yang tertentu. Seterusnya, untuk merumuskan satu shampoo yang jelas, ekstrak herba telah bercampur dengan bahan lain seperti methyl paraben dan EDTA untuk membentuk formulasi baru. Shampoo tersebut telah diuji dengan menggunakan ujikaji pH. Pemalar tindakbalas k dapat dilihat dengan bahawa saponin menurun apabila pertambahan suhu dan juga sonikasi masa dan nilai k diantara kadar 0.1091 sehingga 0.430. Maka, kandungan saponin yang berkepekatan tinggi digunakan sebagai bahan utama dalam melakukan formula bagi shampoo yang asli.
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\( g \)  gram
\( ^\circ C \)  degree celcius
\( y \)  area under the graph
\( m \)  slope of the graph
\( x \)  concentration of saponin
\( k \)  rate constant
\( t \)  sonication time
\( T \)  temperature
\( S_o \)  total content of extractible compounds
\( S_t \)  remained extractible compounds after extraction time
CHAPTER 1

INTRODUCTION

1.1 Background of Study

*Sapindus Emarginatus* commonly known as soapnut is a medium sized tree found in India. It is a genus of about five to twelve species of shrubs and small trees in the Sapindaceae, native warm temperature to tropical regions in both the Eastern and Western Hemispheres. In addition, it is distributed in Indian Peninsula, chiefly in South India. Members of these genus also called “soapberries” because of its pulp have special properties which can produce soap. In addition, it is also an economically important tree which has been brought under silvicultural practices (Troup R, 1921). Based on its special properties, inside from their pulp contain important bioactive compound like saponin to produce shampoo. It also reported that high content of saponin inside pericarp (The Wealth of India, 1972; Gupta and Ahmed, 1990). From its pericarp, two Pisicidal triterpenoid saponins, acetylated triterpene saponins, hederagenin, sweer acyclic sequieterpene glycoside and Mukurozioside IIb15 have been isolated (Wilawan,Pittaya et al. 1990). The soapnut extracts contained 11.58-19.58 % of total saponin which increase importance of soapnut ( Battal, 2002)
Sapindus emarginatus contain saponin, the natural washing ingredient which are likely to soap. When the shells of the soapnut have some contact with water, the saponin will be released. The major commercial sources of saponin are *Yucca schidigera* and also *Quillaja saponaria*. Saponin has a large family which containing a steroidal or triterpenoid aglycone that linked to one or more oligosaccharide moieties. *Sapogenin* or *genin* is the aglycone or non saccharide portion that contain in saponin molecule. This saponin can be divided into three major classes which are Triterpene glycosides (C\(_{30}\)), Steroid glycosides (C\(_{27}\)) and also Steroid alkaloid glycosides (Hostettmann and Marston, 1995). Steroid glycosides saponins are shown in Figure 1.1.

![Molecular structure of steroid glycosides saponin](image)

**Figure 1.1**: Molecular structure of steroid glycosides saponin

The definition of saponin is through their surface activity which has detergent properties. This is because they contain both water-soluble and fat soluble components. They consist of a fat-soluble core, either a steroid or triterpenoid structure, with one or more side chains of water-soluble carbohydrates. They are also strongly surface active and can form stable foams which act as emulsifying agents and form detergent (Hostettmann and Marston 1995). Saponin can be found in many plants especially certain desert plants. It is present in the small amount in some type of food and peas. It is advisable not to consume a large quantity of food that contains saponin. It is more toxic for some other creatures such as fish (Chevallier, 1996). Phytochemicals is chemical that presents naturally in plants. The
phytochemicals saponin have spectrum which can act as antifungal and antibacterial agents and also inhibition of cancer cell growth. Recently, there has been numerous reported on the application of high power ultrasound in the extraction of various phytochemicals such as alkaloids, flavonoids, polysaccharides, proteins and essential oils from various parts of plant and plant seeds. (Hamburger, 1992)

Ultrasonic is sound that ranging from 20kHz to 1 GHz which generated by transducer that can converts electrical energy into high frequency vibrations (Sun and Wands, 2008). Ultrasound assisted extraction is a technique for extract an important compounds from vegetal materials (Vilkhu, et al, 2008). It is also can be used either small or large scales (Vinatoru, 2001). If this technique is compared with other extraction technique such as microwave assisted extraction, it is more cheaper and also the operation is much easier (Chen et al., 2008; Wang & Weller, 2006). The ultrasonic cleaning bath and ultrasonic probe system is the general ultrasonic devices (Vinatoru, 2001; Luque-García & Luque de Castro, 2003). The effect of mechanism by using ultrasound will give greater penetration towards membrane walls, the contents of cell easy to release and also will improve mass transfer for the compound (Kiel, 2007). In addition, it can be carried out at lower temperature to avoid extracts become damage in higher thermal and also loss of volatile components.

1.2 Problem Statement

Nowadays, chemical shampoo is hair care product that consists of dangerous chemical compound such as Sodium Laureth Sulfate that will give long term effect towards our body. Thus, the problem for this research is to replace the dangerous compound from existing hair shampoo with a bioactive compound in herbal material such as saponin. The bioactive compound usually extract by soxhlet method with ultrasonic assisted to reduce time consuming of extraction process.
1.3 **Objectives of Study**

The objective of this research is to extract saponin from *Sapindus emarginatus* using ultrasound assisted extraction in order to formulate natural hair shampoo.

1.4 **Scope of Study**

There are some scopes which need to be focused in order to meet the objectives.

1.4.1 To study the optimum amount of extraction yield from saponin by using different operating conditions such as size of particles, sonication time and temperature.

1.4.2 Test the formulate hair shampoo in term of Ph

1.4.3 To study the kinetic of extraction in term of k (rate constant) value.

1.5 **Rationale and Significant of Research**

The new formulation of natural hair shampoo from *Sapindus emarginatus* can give big impact in pharmaceutical and cosmetic industries. Most of shampoos contain hazardous compounds such as Sodium Laureth Sulfate. By using plant material, it will produce natural shampoo which will replace the dangerous ingredient. Other than that, this type of shampoos is non toxic and good for health. Thus, the shampoo will be safer to be used regularly towards ourselves.

The extraction of bioactive compound from natural plant such as soapberries will give source of saponin which is can be found in every plant material. The extraction by using ultrasound assisted extraction, it will produce higher amount of yield. Besides that, this type of extraction will have lower operating cost. Thus, it will reduce the cost for industrial.
CHAPTER 2

LITERATURE REVIEW

2.1 Plant Material

2.1.1 Plant Description

*Sapindus Emarginatus* is fairly large, deciduous tree which are usually up to 12 m in height, with a globose crown and rather fine leathery foliage. Its leaves in average 30 until 50 cm long, alternate, commonly its narrowly bordered and often slightly falcate. These trees have flower that’s inflorescence a compound terminal panicle in length 30cm with pubescent branches. For its fruit, it is a globose that has 1 seeded drupe or sometimes 2 drupels together. It is in diameter 0.8-1.3cm that smooth, black color and also loose in dry fruit. This fruits have so many names depends which countries it has been planted such as sapindus rarak, sapindus mukorras and etc. It also has bioactive compound in these fruits which is saponin that used to formulate natural hair shampoo in this research. This tree also is multifunction as all part can produce variety of products as shown in Table 2.1.
Table 2.1: Product from parts component in soapnut trees

<table>
<thead>
<tr>
<th>Products</th>
<th>Part of plant</th>
<th>Explanation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Food</td>
<td>Seed</td>
<td>Prepare industrial protein from globulin fraction.</td>
</tr>
<tr>
<td>Fodder</td>
<td>Leaves</td>
<td>Fodder is used for cattle</td>
</tr>
<tr>
<td>Essential oil</td>
<td>Seed</td>
<td>Essential oil produce</td>
</tr>
<tr>
<td>Medicine</td>
<td>Fruit and seed</td>
<td>As cure for epilepsy. From seed, it is used to stop the dental caries and also considered haemolytic.</td>
</tr>
<tr>
<td>Others</td>
<td>Main part</td>
<td>Use as a substitute for shampoo.</td>
</tr>
<tr>
<td></td>
<td>Pulp</td>
<td></td>
</tr>
</tbody>
</table>

2.1.2 Benefit of Sapindus Emarginatus

Traditionally, *Sapindus emarginatus* is used as an anti-inflammatory, antipruritic and also have been used to purify the blood. Its seed are crushed to make an effectives and environmentally friendly natural soaps. In other words, their seed are intoxicant and its fruit has an oxytropic action. Its powder also used as nasal insufflations. Their powdered are said to posses insecticide properties. It cleanses the skin of oil and even use as cleanser washing hair and hair tonic, and forms a rich, natural lather. *Sapindus emarginatus* also has showed that it is a strong anti bacterial activity against the tested bacterial strains (Nair R, 2005). In addition, their pericalp has been reported that contain high content of saponins (Venkatesh V & Sharmal JD et al,2002).

2.2 Saponin

Saponin is a bioactive compound that can be found in plant herbs. It has been reported that saponins presents in fruits which have higher chances including these fruits.
For any type of sapindus, it has four acylated saponins with the main a glycone, hederagenin which is triterpene-type sapogenin have been identified by Hamburger et al in 1992. Triterpene-type saponins series need to be isolated and characterized in other species of sapindus such as S.saponaria (Lemos et al.1994) and S.emarginatus (Kachanapoom et al., 2001). Actually, saponin are easily to find in plant herbs which are group among anti-nutrionally which may caused photosensitization (Flaoyen and Wilkins, 1997: Meagher et al., 2001: Pirez et al.,2002). In others findings, it has shown that saponin has beneficial effect towards animal and also environment by reducing amount of methane produced by the animals (Wallace et al.,2002; Hess et al.,2003 a.b). It also can decrease or eliminate protozoan in the rumen without inhibiting bacterial growth by interact with cholesterol in membranes of eukaryotic cells. In addition, saponins are phytochemical compound that linked towards each chain on sugar chains. In recent years, these type of phytochemical compound will be increase their yielding by using ultrasonic (Z.Hromadkova, 2003).

2.3 Extraction Process

2.3.1 Ultrasonic assisted Extraction (UAE)

Ultrasonic assisted extraction is one of the possible methods to extract the Sapindus Emarginatus. This method is using ultrasound device of ultrasonic which are offers great potential in the processing liquids and slurries. This is regarded as a new way of technology in food, chemical and also pharmaceuticals industries (M.Vinatoru, 2001). It is represent a clean way to accelerate and improve mass transfer process. The function of high power of ultrasound for bioactive compound has been reported by many researchers (Bruni et al, 2002;Albu et al,2004; Wang and Weller,2008). For the enhancement of extraction efficiency of organic compound using this technology is attributed to a phenomenon called cavitation. Cavitation is formation of bubbles of a liquid in a region where the pressure of a liquid falls below its vapor pressure. When ultrasound is applied, a large amount of bubbles formed in liquid medium. When agitated by intense ultrasound, they oscillate and undergo explosive growth and subsequently collapse (Leighton, 1994). The reason collapse because
of limited “space” for them to expand which are bubbles can reach thousands of bar in pressure and Kelvin in temperature. Extraction can be significantly improved with the aid of an ultrasound wave.

Even though the effects of ultrasonic have been studied in over hundred years of herbal species such as ginseng, tobacco but it effect on the UAE on this sapindus emarginatus has not been discovered.

2.3.2 Traditional Extraction

Extraction system is a separation process either solid or liquid. It has several examples of traditional extraction. Soxhlet extraction is one of example of traditional extraction that has been used long time ago and until now. It is required where the desired compound has limited solubility. This kind of extraction is very time consuming and also required a large amount of solvent.

If the desired compound has a significant solubility in a solvent, then a simple filtration can be used to separate the compound from the insoluble substance. Normally, a solid material contain some of the desired compound placed inside a thimble that make from thick filter paper that loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor placed onto a flask containing the extraction solvent.

The solvent is heated to reflux. The solvent vapor travels up a distillation arm, and floods into the chamber housing the thimble of solid. Slowly, the chamber containing the solid material that fills with warm solvent. When the Soxhlet extractor almost full, the chamber automatically emptied by siphon side arm, with the solvent running back down to the distillation flask. The Soxhlet extractor apparatus are shown in Figure 2.1.
For the past 126 years, Soxhlet extraction has been the most respected among all other conventional techniques (M. Itelliar et al, 1999). It serves a dual purpose of extraction step for the isolation of phyto-constituents and as a well established model for the comparison of new extraction alternatives. One of the major significant shortcomings of Soxhlet extraction is the lengthy extraction time that can be 8, 16, 24 hours or more (Pastot, 1997), which results in consumption of considerable time and heat energy. The lengthy time requirement makes it more labor-intensive and limits the number of samples that can be processed which may not be entertained from commercial aspects. Use of large amount of organic solvents requires an additional recovery step and subsequent evaporation to environment.

Figure 2.1: Soxhlet Extractor Apparatus
2.3.3 Differentiate between UAE and Soxhlet Extraction

The differences between UAE and Soxhlet extraction are given in Table 2.2.

Table 2.2: Difference between UAE and Soxhlet extractor

<table>
<thead>
<tr>
<th>Difference</th>
<th>Ultrasonic assisted extraction (UAE)</th>
<th>Soxhlet extraction</th>
</tr>
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<tr>
<td>Process</td>
<td>The sound ranging from 20 kHz to 1GHz which generated by transducer that converts mechanical or electrical energy into high vibrations. (Sun &amp; Wands, 2008)</td>
<td>Soxhlet extraction used to extract soluble compounds from plant material.</td>
</tr>
<tr>
<td>Power used</td>
<td>20kHz</td>
<td>None</td>
</tr>
<tr>
<td>Equipment</td>
<td>Ultrasonic cleaner</td>
<td>Combination condenser and soxhlet</td>
</tr>
<tr>
<td>Time consuming</td>
<td>Less time</td>
<td>Longer time</td>
</tr>
<tr>
<td>Amount of solvent</td>
<td>Less solvent</td>
<td>Many solvent</td>
</tr>
<tr>
<td>Amount of yield</td>
<td>Higher yield</td>
<td>Less yield</td>
</tr>
<tr>
<td>Difficulties level</td>
<td>Lower</td>
<td>Higher</td>
</tr>
<tr>
<td>Cost</td>
<td>Lower</td>
<td>Higher</td>
</tr>
<tr>
<td>Environmental</td>
<td>Friendly user</td>
<td>Friendly user</td>
</tr>
</tbody>
</table>

From Table 2.2, it shows that UAE has more advantages than soxhlet extraction. This is because of time consuming for UAE is faster than soxhlet which required longer time to extract the bioactive plant. Other than that, UAE only need less solvent otherwhile soxhlet need many amount of solvent. Both of techniques are friendly users which not harm to the environment. Therefore, UAE was used to extract bioactive compound from plant material.
2.4 Mechanism in Ultrasonic assisted extraction

Ultrasonic has been used for years in research and also diagnostics which are turning this laboratory based prototype technology into fully operational processes such as food and cosmetics industries. From higher sound, it can convert mechanical or electrical energy into high vibrations (Sun & Wands, 2008). Thus, it will provide some unique condition to derive chemical reactions. These unique conditions will derive from acoustic cavitation which are have sound induced growth and collapse of micrometer sized cavities in a liquid. Through these condition, it will result such as emission of light and dissociation of chemical bonds. Thus, it will refer as sonochemistry.

Sonochemistry is about the effect of sonic waves and wave properties on chemical systems. The chemical effect is indirect interaction with molecular species. Some studies shows that no direct coupling with chemical species on molecular level. It is arises from acoustic cavitation which the formation, growth and implosive collapse of bubbles irradiated with sound. Cavitation is a process where mechanical activation destroys the attractive forces of molecules in the liquid phase. When sonicating liquid at high intensities the sound wave will propagate into the liquid media result. By applying the ultrasound, compression of the liquid is followed by the rarefaction which a sudden small pressure drops form, oscillating bubbles of gaseous substance.

From Figure 2.2 shows that the bubble collapse in liquid phases will produces enormous amounts of energy from the conversion of kinetic energy of the liquid motion into heating the contents of the bubbles. The compression of the bubbles during cavitation is more rapid than thermal transport which generates a short-lived localized hot spot. During the implosion very high temperature and pressure are reached locally. The implosion of the cavitation bubbles also results in liquid jets of up to 280 m/s velocity. The resulting shear forces break the cell envelope mechanically and improve material transfer. The ultrasound can have either destructive or constructive effects to cells depending on the sonication parameters employed.
When liquids contain solids, similar phenomena may occur with exposure to ultrasound. Once cavitation occurs near an extended solid surface, cavity collapse is nonspherical and drives high speed jets of liquid to the surface. These jets and associated shock waves can damage the now highly heated surface. The liquid powder suspensions produce high velocity inter particle collisions.

The extraction of bioactive compounds from seed classically based upon combination of solvent, heat and agitation. This significantly improved by using the high power of ultrasound which are energy that has been generated from collapsing bubbles. This collapsing bubble provides greater penetration of the solvent into the cellular material and also will improve mass transfer (Vinatoru, 2001; Vilkhu, Mawson & Bates, 2006). At higher ultrasonic intensities, this extraction process can be improved by the disruption of cell walls and thus it will release the bioactive components. From previous journals, it has studied about the effect of ultrasonic on supercritical extraction of ginger. Balachandran (2006) states that both rate and their final yield have been improve significantly. The cavitation events in a supercritical fluid seem impossible due to absence of gas and liquid boundaries. Otherwhile, Carcel and co-workers showed that the intensity of brine solution into meat proportional to the applied intensity. The chemical effect of ultrasound

**Figure 2.2:** Implosion of bubbles
irradiation does not depend on the molecules present to the collapsing cavity, it also depend on the magnitude of the temperature rise.

Temperature will affect the vapor pressure, viscosity of the liquid and also surface tension (Muthukumaran et al, 2006). When temperature keep increasing, the number of cavitation bubes also will increasing, thus the higher vapor pressure will dampened. At the higher viscous, the cavitation bubbles will be less. So when the temperature increased, the viscosity will decrease and it will allow more violent collapse. Thus, there will be an optimum temperature at which to form enough violent cavitation bubbles to avoid the dampening of vapor pressure.
CHAPTER 3

METHODOLOGY

RAW MATERIALS, CHEMICAL & REAGENT, AND APPARATUS PREPARATION

EXTRACTION

ULTRASONIC ASSISTED EXTRACTION

SAPONIN ANALYSIS

FORMULATION OF SHAMPOO

TEST OF SHAMPOO

Figure 3.1: Procedures of experiment
3.1 Experimental

The flow chart for describing overall experiment is shown in Figure 3.1

3.1.1 Raw Material, chemical & reagent and apparatus preparation

Dry *Sapindus Emarginatus* used in this study were collected from India. All samples have been washed and dried under the shades for 14 days (Mali et al, 2010). The dried of samples were sliced into smallest pieces. Then, it need dry until no moisture in their shells. Next, these drying shells were ground into fine powder before extraction. The picture of soapnut is illustrated as in Figure 3.2.

![Figure 3.2: Sapindus Emarginatus seed](image)

**Table 3.1:** List and purpose of chemical used

<table>
<thead>
<tr>
<th>Purpose</th>
<th>Chemical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isolation of saponin</td>
<td>Methanol, Butanol, and Diethylether</td>
</tr>
<tr>
<td>HPLC analysis</td>
<td>Methanol (HPLC grade), Acetic acid (HPLC grade), Saponin standard</td>
</tr>
<tr>
<td>Formulation of shampoo</td>
<td>Xantham gum, EDTA, Methyl paraben, Orange oil</td>
</tr>
</tbody>
</table>