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Full Length Research Paper

Formulation of herbal conditioner shampoo by using extract of fenugreek seeds and evaluation of its physicochemical parameters

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Nowadays, people are interested in hair preparations and conditioner materials, such as shampoos. Hair tonic and conditioner formulations containing herbal extracts, such as fenugreek, can prevent hair loss and retain hair conditioning. First of all, the proved seeds of fenugreek were extracted with 50% ethanol by maceration method, and then were freeze dried and stored in the fridge. After preparing the formulation, some physicochemical properties such as pH, foam formation, viscosity, conditioning and wettability were evaluated. The pH of the formulated shampoo was determined as 6.6. The results of its rheogram showed good thixotropy property. High foam production and stability were observed; this may be due to the existence of saponin in fenugreek seeds extract. On the basis of wettability and conditioning results, formulated shampoo can represent an attractive and suitable product. The pH of the formulated shampoo was in the standard range of 6 to 8. The formulated shampoo has also shown the better foaming productivity and thixotropic properties which shows its suitable viscosity. The wetting effect of shampoo was taken 5 min which indicates its proper quality in comparison to some other shampoos in the market. Based on the wettability and conditioning data, it can be concluded that the formulated shampoo has a good quality of introducing it to the market.

Key words: *Trigonella foenum graecum*, herbal shampoo, conditioner, fenugreek.

INTRODUCTION

Shampoos are kind of formulation that are used for hair and body washing or therapeutic purposes. Shampoos are expected to be much more than mere cleansing agents. Shampoos have many properties in addition of their detergency, such as conditioning and hair shining. They are expected to be non-irritating to skin and mucous membranes (Hilda, 1996; Trüeb, 2001; Feldman and Yentzer, 2009). There are many different varieties of ingredients for making a proper shampoo. Each of these ingredients have special role in shampoo's formulation. The major ingredients used in making a shampoo are detergents (surfactants), conditioning and active ingredients for hair manageability, additives that modify the surfactant effect (viscosity control agents, foam stabilizers and viscosity modifiers), stabilize the product (preservatives and anti-oxidants) and increase its appeal (fragrances, essence, anti-UV light protector, dyes and ingredients for consistency and a pearlescent appearance). Some of these additives are arbitrary while many of them have to be added in a shampoo formulation to increase its stability and safety (Madani et al., 2011; Trüeb, 2007; Jeffries, 2005; Wolf et al., 2001). Conditioning agents are examples of these additives. They have been attractive components in the recent years. Surfactants are specific conditioners but there are many other materials used as conditioners, such as paraffin and lanolin. There are also many other

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materials that serves as conditioners like peptides, egg derivatives and synthetic resin. These materials were added in shampoo formulation as hair conditioner and hair shining agents. Other polymers used in the formulation of shampoos that are capable of drawing crisp hair could be water soluble phosphate salts and amino ethyl ester poly acrylic acid. The resin is used in the formulation of shampoos containing surfactants. Another compound called Mirapol A15 could be seen as a conditioner in the market and it has shown desirable properties, such as anti-static electricity (Trüeb, 2007; Bellare et al., 2001; Berthiaume, 1997). Water soluble proteins, such as hydrolyzed collagen with molecular weight between 500 to 10,000 Dalton and their tetravalent derivatives cause improve in the ease of wet and dry hair combing. These compounds have protective effects for hair and make them softened and possible stimulatory effects of shampoos. This combination causes hair softening and gives hair status like silk mode. In order to smooth the hair and make them glow the oily materials called super fatty materials are used. This group of materials can be lanolin ethoxylated derivatives, silicone oils, liquid paraffin, sesame oil and Zhvzhvb oil. The recent oil is produced from *Simmondsia californica*. These materials are deposit in the hair's keratin and act as slippery agent (Hilda 1996; Nehdi, 2011; Cerchiaraa et al., 2010).

Discussion about hair products is highly regarded nowadays. Hair tonic and conditioners are formulated as shampoos contain a large number of cosmetic products (Wu et al., 2010). They may have herbal extracts like fenugreek extract. The fenugreek extract has important role in reducing hair loss and its conditioning mode (Wichtl, 1994). Therefore, this study was designed to formulate shampoos containing fenugreek extract and its physicochemical properties were studied.

MATERIALS AND METHODS

Plant extraction

Plant extraction was performed by maceration method. First, 200 g of the plant was weighted and it was extracted using ethanol (50%) within 72 h. The extract was smoothed every 24 h and the extraction continued using a new alcoholic solvent. Collected extracts was concentrated below 50°C by rotary evaporation method and it was then solved in water and concentrated finally by freeze drier. The produced powder was then weighed and stored with a proper absorbent in the fridge (Wallis, 1985).

Formulation of a conditioner shampoo

To formulate a basic shampoo; definite amount of sodium luryl sulfate, sodium sulfosuccinate, N-alkyl betaein and coconut fatty acid diethanol amid were added to an aqueous demineralized water solution containing propyl and butyl paraben (1%). Sodium luryl sulfate and sodium sulfosuccinate with coconut fatty acid diethanol amid were added into demineralized water and were mixed gently to avoid making any foam. Then, propyl and buthyl paraben were added and mixed gently. Previously prepared fenugreek extract was added to the basic shampoo formulation, after which it was mixed gently and then topped up with water where needed (Berthiaume, 1997; Barel et al., 2009; Martinsa et al., 2011).

Evaluation of physicochemical pH assessment test

The pH of a basic shampoo (formulation without extract) and shampoo with extract were measured by pH meter. The measurements were performed in triplicate and mean values and standard deviation (SD) were used for analysis. The experiment was performed in 1% shampoo solution at 25°C (Kumar and Mali, 2010).

Foam productivity determination

10 ml of shampoo was rotated with a certain speed in a graduated cylinder by Erveka machine for 2 min. The foam volume was measured at 0, 1, 4, 16 and 24 h (Barel et al., 2009; Kumar and Mali, 2010; Arzhavitina and Steckel, 2010).

Moisturizing time determination

One gram hair ball with approximate of 20 cm^3 size was placed on the surface of 60 ml of different dilutions of shampoo and the complete sinking time of the ball hair in the shampoo was measured (Barel et al., 2009; Kumar and Mali, 2010; Lodge and Bhushan, 2006).

Rheology experiment

Rotational spindle Brookfield viscometer (Model DV-I Plus, LV, USA) instrument was used for rheology experiment (Sinko, 2006; Al-Achi et al., 2007; Abu-Jdayil and Mohameed, 2004).

Conditioning effect experiment

In order to test the conditioning effect of the shampoo, we had to see how it is easy to comb the hairs, and to do so, we had to use a comb connected to a spring and a scaled page. The scaled page was able to display the rate of hair resistance against combing. In this method, the incoming force on ergo-meter caused by moving of the comb between hairs after and before using of shampoo was measured. This experiment was done five times (Barel et al., 2009).

Study of detergency activity

In order to make this test, the artificial sebum formula was used as follow: olive oil (15%), coconut oil (15%), stearic acid (15%), oleic acid (15%), paraffin wax (15%) and cholesterol (15%). The experiment was performed as follow: 3 g of hair was placed in 20 ml of sebum solution (10%) in hexane for 15 min and the mixture was stirred in this period. The hairs were then taken out from the mixture and hexane was evaporated and the dried hairs at room temperature were weighted. The 3 g hair was divided into two equivalent portions. One portion was used for the experiment and the other one as control. The first portion was then washed with 0.1 ml tested shampoo (10%) and was dried. To make sure

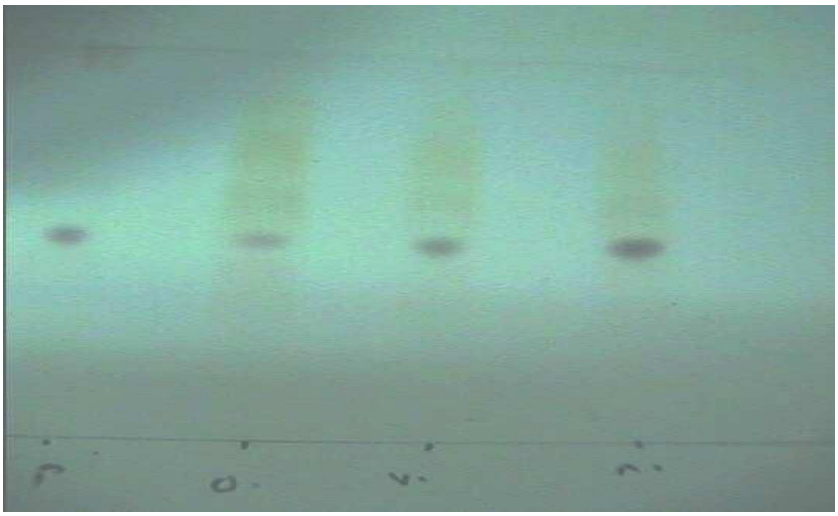

Figure 1. The TLC results of fenugreek extract.

Table 1. Determination of shampoos pH.

Product	pH ± SD*
Basic Shampoo	6.57 ± 0.11
Shampoo containing fenugreek extract	6.22 ± 0.20

*The results are from three times measurements (Mean ± SD).

the hairs are dried completely, we had to store them in oven (60°C) for 4 h. The remained sebum was then mixed with the hairs used for the experiment after shampooing and it was also mixed with the hairs for control experiment without shampooing in hexane. The hexane was then evaporated within 30 min and the extracted sebum from both portions was measured on the basis of its weight percent. The cleaning percent was calculated according the following formula (Mainkar and Jolly, 2000; Hamel et al., 2011; Glassman, 1997):

Removed sebum after using shampoo (Detergency percent) = 100 - (T+100/C)

where T is sebum weight in hairs portion for the experiment and C is sebum weight in hairs portion in control sample.

Consumer reviews

In order to study the consumers view, twenty four healthy female subjects between the age of 18 and 38 with no apparent hair or dermatologic disease and hair of sufficient length such that it could be combed were selected and finished the 2-week double-blind cross-over study to evaluate the effect of two different shampoos. Subjects were asked to shampoo a minimum of three times weekly, after a one week washout period with a basic non-conditioning cleansing shampoo (Nivea energy shampoo, Beiersdorf AG, Germany) to standardize hair condition prior to study initiation. No conditioners or other shampoos were allowed during the washout period. Subjects were then randomized to receive one of the two study shampoos to use for one week. Twelve subjects received the novel conditioning shampoo and the other subjects received base shampoo. At the end of one week, subjects crossed over to use the other study shampoo for an additional week. At the end of the second study week, subjects were asked to pick their preferred shampoo for continuous use in the last two weeks of the study. The appropriate volumes of shampoo in similar packing were delivered to the applicants. Self-assessments demonstrated hair benefits after three uses. The evaluation forms were given to the consumers and their views were considered. The questionnaire contained many questions, including how it is easy to comb the hair, visual breakage, softness, shine, scalp itching and dryness, lack of static electricity, ease of rinsing, foam volume, viscosity and cleaning. The applicants' views were then analyzed (Churchill et al., 2009; Draelos et al., 2005).

RESULTS

Thin layer chromatography (TLC) of dried extract was performed in a solvent system including (First solvent: water, methanol and ethyl acetate with 1, 1.35 and 10 ratio, respectively. Second solvent: water, acetic acid, methanol and n-bothanol with 1, 1, 1.5 and 4 ratios, respectively). Plates were then studied under 254 nm UV using two reagent (The first reagent: 1 g iodine with 1 g potassium iodine was dissolved in 100 ml ethanol. The second reagent: 25 ml of 25% HCl with 25 ml of 96% ethanol were mixed) in order to investigate the existence of trigonelline (Figure 1). The pH results are as shown in Table 1. The result of foam activities are as shown in Figure 2 and wetting time experiment

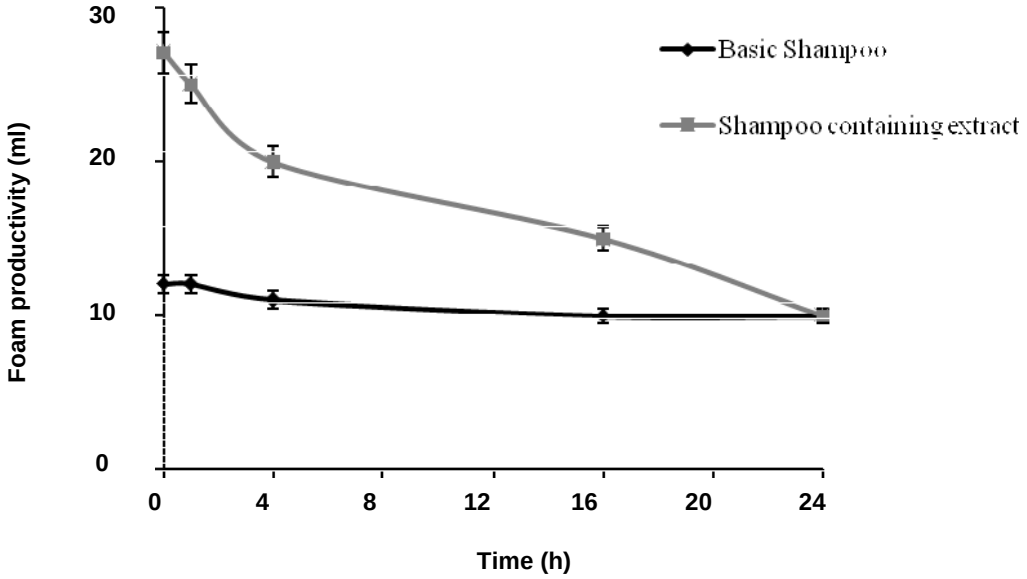

Figure 2. Foam productivity (ml) at different times (Mean ± SD).

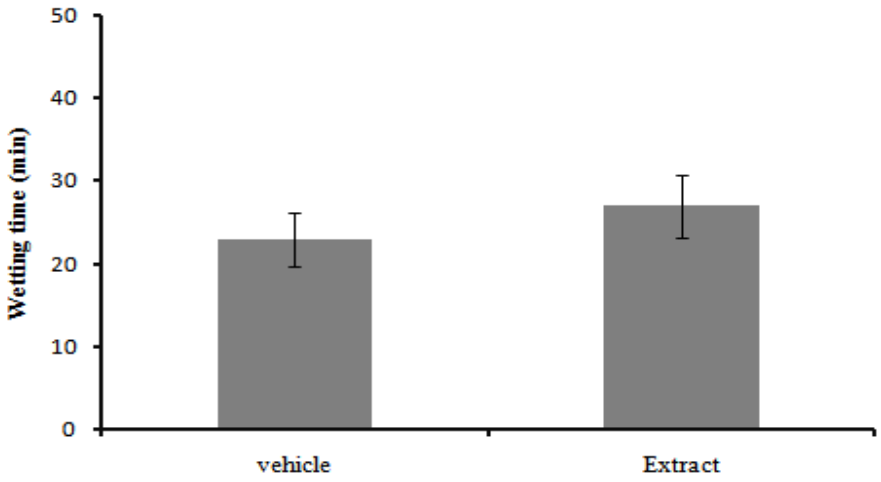

Figure 3. Wetting time results; the results are from three times measurements (Mean ± SD).

results are displayed on Figure 3. In order to perform rheology experiment, a viscometer machine was used. The rheogram is as shown in Figure 4. The conditioning experiment was done and the results of the force rate before using the shampoos was reported after ten times repetitions and the average calculated. The results of detergency are as shown in Table 2. The satisfaction of consumers after using the shampoo and its results are as shown in Figure 5.

DISCUSSION

Recent studies show that hair loss and its related disorders can have profound psychological effects and affect people confidence. In 2003, it has been reported that about $ 25 billion dollars is spent on hair care in the United States annually. There is a remarkable number of non approved products for treating hair problems available in the market without any scientific evidence indicating that they are effective products (Newall et al., 1996; Loretz et al., 2006; McNamara et al., 2007).

Shampoos pH should be between 6 and 8. Many studies have been made to make the shampoo's pH to the keratin isoelectric point. Studies show that there is not significant link between sensitivity and pH. It was reported that the application of shampoo with pH more than 8.7 or less than 6.6 is not idealistic, because

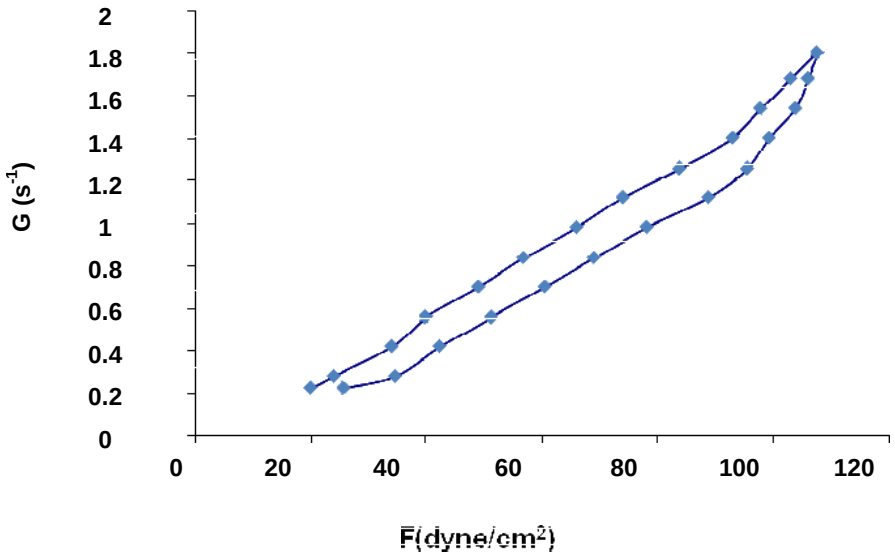

Figure 4. Changes of shear speed versus shear stress in shampoo containing of fenugreek seed extract.

Table 2. Removing of sebum using different shampoos (%).

Shampoo type	Removing sebum (%) ±SD*
Basic shampoo without extract	72.3 ± 0.8
Produced Shampoo	75.7 ± 0.1

*The results are from three times measurements (Mean ± SD).

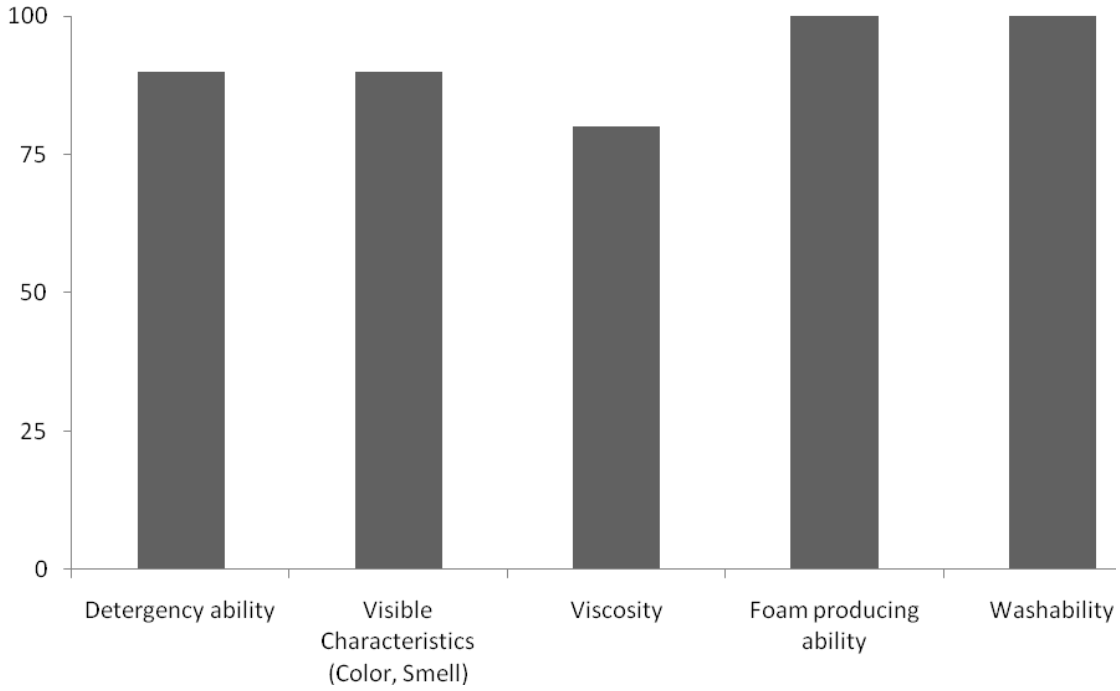

Figure 5. Consumer satisfaction (%) about different characteristics of produced shampoo.

ophthalmic solutions are used in this range, without discomfort and conjunctival irritation (Mainkar and Jolly, 2001). The pH of the shampoo with extract was 6.22 ± 0.20 which is more appropriate as compared to the pH of shampoos available in the market. This pH is a proper pH for shampoos and it makes a proper electric charge for hair conditioning (Lenoir, 2011). The environmental pH has effect on the absorption of proteins on hairs. For instance, there is a higher absorption in pH 6 to 9. Hairs become softer in the presence of conditioners for pH of 6.2. According to this, our product has appropriate and proper conditioning status (Berthiaume, 1997).

The consumers often give a lot of attention to the strength properties of foam productivity and this is one of the favorite factors for consumers. This research showed that our product has more foam productivity as compared to similar product in the market. The high volume foam productivity is usually considered as shampoo efficiency. The foam produced by a shampoo has psychological effects and it is also a value to show that shampoo has sufficient ability for hair cleaning (Arzhavitina, 2010). In this case, many other factors, such as the time of foam productivity, foam consolidation and its stability should be considered too. Foam stability prevents replacement of removed germs from the hair. In the case of the foam subsidy, some of the suspended objects deposit on the hair again. In this case, the hairs become opaque. Hence, our new formulation could prevent replacement of germs and soil on hairs due to its more foam productivity and stability than its similar products. According to consumers view, this formulation has no visual sensitivity and it produces a reasonable amount of foam. This study showed our product makes plenty foam and it has good stability. It was also shown that hairs rinsing was very convenient after using our product (Hilda, 1996; Barel et al., 2009; Arzhavitina and Steckel, 2010).

Viscosity and rheological properties play important role for liquid shampoos. They could influence many of the product attributes, such as shelf-life, its beauty, its transparency, easy removal from its packaging, its expansion and its consistency (Al-Achi et al., 2007). The rheological properties of this formulation showed that this formulation has a proper trait. In another word, it has a proper viscosity and if it is exposed to certain cutting speed, shear stress viscosity of the system will be reduced over time consequently. This means that the products go out from the container easily. In these systems rheogram, if the descend curve is located on the left side of the ascend curve, it means thixotropy property is ruled, because after the cessation of stress it takes some time to recover its viscosity. The more the surface size below the thixotropy graph the more the thixotropy property; in another word, after the cessation of stress it takes longer time for the system to recovers its viscosity. In other words, in the thixotropy systems, because of force engagement, the changing is from gel to pannier mode (Abu-Jdayil and Mohameed, 2004). In rheological behavior, thixotropy systems are usually formed from non-symmetrical particles or large molecules that are capable of forming many secondary bonds between themselves and a relatively strength three-dimensional structure. This structure has a gel mode in respite status which is important for the consumers due to psychological reasons. This status prevents the pouring of the shampoo from the head down. In cosmetics products, macromolecules are opened more when compared with their relief when the tension and flow are increased. This causes decrease in their viscosity. However, our rheogram formulation seems more appropriate in comparison with the samples taken from the market (Sinko, 2006; Al-Achi et al., 2007). As we did not use any viscose substances for making our shampoo, it showed less thixotropy than the samples taken from the market. According to a general formula for shampoos, the amount of viscose substances in shampoos is up to 10%. As fenugreek seed extract contains viscose components, we decided not to use any other viscose active material in the formulation and it was observed that the shampoo with fenugreek extract has more thixotropy than the one without the extract, while all other shampoos taken from the market contained viscose components.

One of the important factors affecting the separation of soil from substrate is moistening of the substrate surface and the ability to penetration detergent component in the soil. This reaction continues by reducing the surface tension. This means that the detergent dissolves and removes soils much more when the surface tension is lower and the moisturizer is higher. The moisturizing time is usually depended on the concentration of detergent and other ingredients (Wolf et al., 2001; Mainkar and Jolly, 2001).

This study showed that our formulation has suitability sovereignty of its moisturizing. Therefore, based on the results of strength and moisturizing time and foam productivity, our formulation could easily penetrate hairs and this conformed to the results taken from consumers view. However, it is possible to study about hair coarsening and its tendency for curly mode hair after applying the shampoo in the hair conditioning experiments (Lodge and Bhushan, 2006).

The hair shining and its glittering are two values for consumers showing the suitability after using a shampoo. Since fenugreek seeds have carbohydrates and protein compounds, it is expected that a shampoo containing fenugreek extract makes hair combing very easy. In another word, it makes changes in the surface features and this theory has been confirmed in our conditioning experiments. A group of substances that protect, strengthen and change or modify the hair fiber and they can be used in partial or general application include hydrolyzed protein, especially collagen, keratin

and casein milk. The refreshing and healing effect of hair conditioning products containing protein has been 95%. A similar result has been proved for Rynes® cream which contains 5% of animal hydrolyzed protein (like Lexin from Inolex Group) while Rynes® without any protein had 25% effect. New hydrolysis containing quarterlies amino groups are available now and they are helpful in the strengthening efficiency effects of proteins. Elastin and collagen hydrolysis mixture can make the hair to be soft. Oleic acid, condensed protein and homologous of oleic acid are used for better hair combing and thick hair, respectively (Hilda, 1996; British Herbal Pharmacopoeia, 1989).

Fenugreek seeds also contain proteins and amino acids and this is important for hair strength and its softness. Hydrolyzed proteins are important components in the formulation of conditioning shampoos nowadays. Fenugreek prevents hair loss and it contains lecithin, which helps hair growth. The seeds contain four flavonoids and two steroid saponins. Fenugreek is an effective herb to reduce hair loss and baldness in male. Fenugreek seeds are very nutritious and as a result they are effective in reducing hair loss, baldness and thin hair. They also contain progressive hormones that increase hair growth. There are natural emulsions for hair moisturizing that contains high concentration of proteins and are similar to fenugreek seeds, they have the ability to eliminate hair loss and hair damage. Flavonoids are important for increasing blood cells and they also help the blood to circulate to all parts of the body including scalp. Trigonelline helps the blood cells and it increases the blood flow in the hair follicle. In this study, according to TLC results, 50% fenugreek was used which contains substantial trigonelline. Fenugreek caused blood vessels dilatation in the scalp and thus, increases the blood flow in the area. Researchers have explained that trigonelline or trigonellic acid extracted from fenugreek has strong effect on the skin and animal nails. The reason is because it provokes the epidermal cells in animals to start their growth again especially the hair cells. It contains alkaloid gonylin and other materials, such as biotin, Asian ginseng extract and horse chestnut or tocopherol. Based on the literature, ginseng has enhancing effect on trigonelline and it decreases its action effect from four months to one month (British Herbal Pharmacopoeia, 1989; Mai, 1999; Rechinger and Trigonella, 1984; Mainkar and Jolly 2000; Lee et al., 2010).

Based on the results from consumers view, the formulated shampoo showed that all the consumers were happy for their hair rinsing and 93 to 95 of them were satisfied about lack of static electricity and easy hair combing. According to this research results, our product is able to suspend the soil materials easily. In this study, we also found that our shampoo has strong cleaning effect and has ingredients in formulation such as sodium luryl sulfate and N-alkyl betaine with high cleaning properties. Although, the surface and inter-surface tension properties are used as *in vitro* laboratory techniques for evaluation of shampoo and for selection of detergents, but it should be noted that still there is no known substitution for the experiments that should be done on applicant's hair. The main reason for this is that the effect of hair washing after using the shampoo cannot be rebuilt with *in vitro* experiments, but the comparable experiment can be done. Some factors, such as separation of grease and soil ingredients from hair after its washing in hard water should be considered as important criteria for a proper shampoo. Those criteria are subjective criteria and they should be considered based on consumers' view (Trüeb, 2007; Wibowo and Ng 2002).

The shampoo conditioning effect assessment is not easy as it depends on the consumer views and the consumer views is also depended on previously used formulations, their expectation from the new shampoo and the new changes that they feel in their hair and skin. It must be noted that conditioned hairs should be soft, shiny, smooth, slippery and easy to comb in wet and dry status. Other properties also are color, odor and physicochemical properties which can be used for comparison when consumers were asked if they have hair properties such as foam productivity, cleansing, sensitivity, dandruff and hair loss (Bellare et al., 2001; Trüeb et al., 2005).

In this study, it was demonstrated that 95% of applicants were happy with hair combing comfort, smooth and shiny hair and lack of static electricity in their hair. In this study, hair conditioning was demonstrated, because of the existence of trigonelline in fenugreek extract. It also showed that our product makes high volume of foam and all consumers showed it in their questionnaires and this is also because of saponin in fenugreek extract. Based on this and other studies, it appeared that the use of herbal shampoo is better than a synthetic shampoo (Cerchiaraa et al., 2010; Glassman, 1997). Some shampoos contain high volume of detergent and it removes more than 80% of oil from hair but there is no such problem in the use of herbal shampoos. It is recommended that it is better to use herbal extract like fenugreek extract rather than using other chemical because herbal shampoos are safer and healthier (Mainkar et al., 2001).

Conclusion

According to the assessment of physicochemical formulation and results taken from consumers, it can be expected that fenugreek conditioner shampoo has the criteria needed for a standard conditioner shampoo. Finally, it is recommended that making standardized fenugreek extract based on the amount of trigonelline and producing its shampoo as industrial products should be

considered. Meanwhile, there is need to do more clinical research in this case.

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