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Shampoos Then and Now : Synthetic versus Natural*#

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Abstract — Hair care has been important to mankind from times immemorial. The market today is replete with synthetic hair care products, pushing natural materials to the background. These natural materials, being produced from plants, are bio-degradable and non-toxic. In a bid to investigate natural products, we studied the surface tension, foaming and relevant parameters of natural surfactants extracted from Shikakai (*Acacia concinna*) and Seto Siris (*Albizia procera*), traditionally used in hair care. In order to get a better understanding, Johnson's Baby Shampoo has also been studied. Shikakai showed very prominent surface tension reduction and high foaming, showing that it can be a potential candidate for good hair care. These effects are not so prominent for Seto Siris. However, as it is widely available in the hills, large quantities can be used. Qualitative measurement of their cleaning properties and subsequent comparison with commercial shampoo show that both plants have a large potential in providing nontoxic natural hair care products.

Keywords : *Natural Surfactants, Foam Formation, Foam Stability, Surface Tension.*

INTRODUCTION

The hair care sector is probably one of the largest sales units amongst the cosmetics. Shampoos are used to cleanse the hair and the scalp [1]. Today the cosmetic market has become extremely competitive, producing various brands in order to catch the customer's attention, each claiming to be better than others [2]. Synthetic detergents used in shampoos available in the market are harmful and toxic; affecting the health as well as the environment [3]. They destroy the environment by releasing non-bio-

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degradable chemicals affecting the aquatic life and algae [4, 5, 6]. These detergents leave chemical residues on clothes, which enter our bodies, either through the skin or lungs, leading to several health problems, including allergies and skin infections [7]. The fragrances used in detergents can also prove allergic and be irritating to the lungs, causing health effects on people with asthma or chronic heart problems [8]. On the other hand, availability and usage of natural detergents is minimal today. Having only natural products, they are not harmful to that extent, either to the user or to the environment.

Natural surfactants or saponins are a group of water soluble glucosides where a hydrophobic group consisting of a polycyclic agylcone called a sapogenin and a sugar chain is joined by an ether bond (Fig. 1).The presence of both polar (sugar) and non-polar (agylcone) groups provides surface active properties in saponins [9]. Saponin occurs in plants as a mixture of structurally related forms with similar polarity comprising of large numbers of surfactants [11].

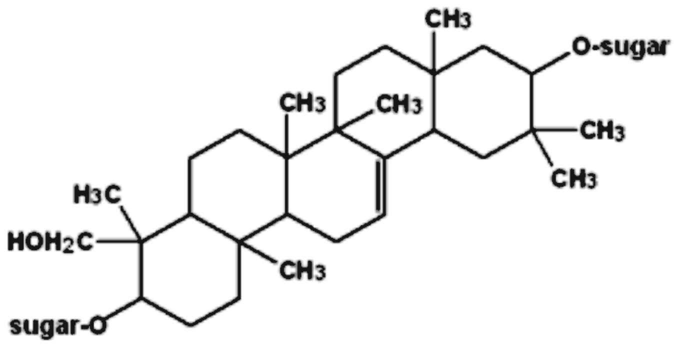

Fig. 1. A generalized structure of Saponin [10].

Shikakai, (*Acacia concinna*) is a climbing shrub found in Asia and central and southern India [12, 13]. Pods of Shikakai have traditionally been used for washing hair. Seto Siris (*Albizia procera*) is a 30 m tall tree found in the sub-Himalayan tracts from Yamuna eastwards to West Bengal, Satpura Range and South India. Its leaves are used as a natural detergent [14]. Both the plants belong to the Fabaceae family. In this paper, the extracts of these plants have been referred to as natural shampoo and compared with synthetic shampoo.

Foam production is an essential criterion to assess a detergent [15]. While the foam properties and detergent abilities of synthetic detergents have been studied extensively, natural detergents have not been well studied. This study investigates foam producing capacity, foam stability, surface tension, wetting ability, dirt dispersion and cleaning ability of solutions of natural detergents extracted from Shikakai and Seto Siris. For comparison, we have also studied Johnson's Baby Shampoo, a synthetic detergent available in the market.

MATERIALS AND METHODS

Natural Detergents :

Plant Collection —

Shikakai pods were bought from Nivedita market, Mahavirsthan, Siliguri, West Bengal, India. Seto Siris leaves were collected from Neeha Busty in South Sikkim, Sikkim, India. The plants have been identified, referred and authenticated by The Ayurvedic Regional Research Institute, Department of AYUSH (Ayurveda, Yoga and Naturopathy, Unani, Siddha and Homoeopathy), Gangtok, Sikkim, India.

Extract Preparation —

The pods of Shikakai and leaves of Seto Siris were washed in water and sun dried. The dried sample was weighed and soaked for one hour in distilled water. The solution obtained was filtered and used for the experiments.

Concentration Calculation —

The concentration of the natural surfactant in the solution could not be calculated easily as the amount of natural detergent that dissolved in water was far less than the amount of sample soaked. The concentration was estimated by calculating the specific gravity of the sample, using a specific gravity bottle.

Let m_1= mass of the empty bottle, m_2= mass of bottle filled with water, m_3= mass of bottle filled with solution, ρ_{water}= density of water at ambient temperature. Concentration *C* is given by,

$$C = \rho_{water} \times (m_3 - m_2)/(m_2 - m_1) \quad (1)$$

Synthetic Detergent :

Johnson's Baby Shampoo was bought from the market with Lot Number BB9087. It was weighed and dissolved in distilled water and diluted as required.

EXPERIMENTAL METHODS

Qualitative Saponin Test :

1 gram of plant extract was boiled in 5 ml of distilled water (conductivity 6.15 ±

0.3 mS/cm) and filtered. 3 ml of distilled water was added and shaken to develop frothing. This was warmed to 45°C. Persistence of froth indicated the presence of saponin [16]. This test was performed in the Department of Microbiology, Sikkim University. The hardness of distilled water was 25 ppm as CaCO3 measured by HiMedia Water Test Kit.

Surface Tension Measurements :

Surface tension of the surfactant sample was measured using KSV 2000 Langmuir Balance Tensiometer with Wilhelmy Plate Method at a room temperature of 25 ± 2°C [17, 18]. These experiments were carried out at Centre for Soft Matter Research, Jalahalli, Bangalore, India.

Foaming Ability and Stability Studies :

Although foam generation is not directly related to the cleaning ability of a shampoo, it is useful to the consumer and hence an essential criterion in evaluating shampoos [15]. In order to understand foam stability, it is necessary to understand the processes taking place in foams, which result its decay.

The liquid present between the bubbles in the foam drains out due to gravity and pressure difference between the film lamella and the plateau borders. This drainage causes the thinning of the lamella and formation of dry foam.

The thinning lamella coalesce producing large bubbles [19, 20]. This occurs when the foam is dry and the film is very thin.

Disproportion in the bubble causes coarsening of foam. The pressure inside a small bubble is higher than that inside a large bubble. This causes the gas in a small bubble to diffuse into a large bubble through the intervening liquid. This phenomenon is called Ostwald Ripening. These three processes are responsible for foam decay.

Fig. 2 show the experimental set-ups used to study foaming ability and stability by Bikerman's Method and Shaking Tube Method.

Bikerman's Method — 40 ml of surfactant solution was taken in a 100 cm long cylindrical column with a fritted glass filter. Nitrogen gas was passed through the solution. Maximum height of the foam column gave a measure of the foaming ability [21].

Shaking Tube Method — 40 ml of surfactant solution was taken in a 100 ml measuring cylinder. The solution was vigorously shaken by hand. The amplitude of shaking was around 5 cm while the frequency of shaking around 3 Hz. The maximum foam height gave the foaming ability.

The decay in the foam height was monitored using a stop watch [22, 23, 24].

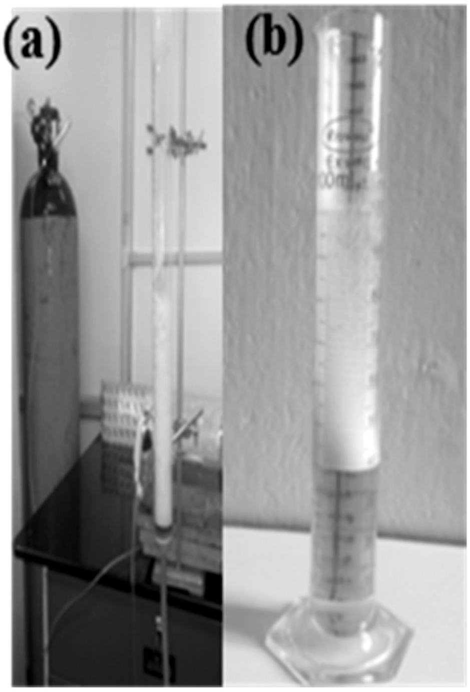

Fig. 2. Experimental set-up for Foaming Ability and Stability by (a) Bikerman's Method and (b) Shaking Tube Method.

The stability of foam can be conveniently expressed in terms of the time taken for the foam column to rupture to half the height ($t_{1/2}$) [25]. However, for some metastable foam the $t_{1/2}$ parameter may run to hours or even days, which makes it impossible to measure. Hence the R5 parameter is more commonly used to express the stability, which is defined as

$$R5 = \frac{h_5}{h_0} \times 100 \tag{2}$$

where h_o = maximum foam height and h_5 = foam height at 5 minutes [22].

Factors Affecting Foaming Properties of Natural Detergents :

The foaming properties were characterized on the basis of parameters like soaking time and soaking temperature in order to optimize saponin extraction. The effect of soaking time was studied by soaking the samples for 24 hours in distilled water. Effect of temperature on saponin extraction was studied by heating the solution to different temperatures after soaking.

Wetting Ability :

Wetting plays a crucial role in the removal of soil, dye and oils. The comparative efficacies of different surfactants are determined by their wetting abilities [26]. To measure the wetting ability, a 5 cm × 5 cm cotton cloth was floated on the solution surface. The time required for the cloth to begin to sink was measured [27].

Dirt Dispersion :

A shampoo is considered to be of poor quality if dirt remains in the foam. The reason is that it is difficult to rinse the dirt from the foam, hence it may re-deposit in the hair [1, 28]. To test the dirt dispersion ability, 1 drop of India ink was added to 10 ml of surfactant solution in a test tube and shaken. The amount of India ink in the foam was estimated [27, 28]. For comparison, we used two bottles with the surfactant solution and added India ink to only one. Both bottles were shaken simultaneously and then the amount of ink in foam estimated.

Cleaning Ability :

The cleaning of dirt is the primary aim of shampoo. This was tested on a 5 cm × 5 cm cotton cloth. The cloth was soaked in water for 24 hours, dried and weighed (W_1). It was then dipped in simulated dirt (1 gm coconut oil and 1 gm paraffin wax dissolved in 100 ml hexane). It was removed, dried and weighed (W_2). The cloth was then placed in the surfactant solution and shaken. It was taken out, rinsed in clean water, dried and weighed (W_3) [29]. The cleaning was calculated using the equation :

$$C = (W_2 - W_3)/(W_2 - W_1) \times 100 \tag{3}$$

RESULTS AND DISCUSSION

Saponin Test :

TABLE 1

Result of saponin test for natural detergents

Name	Foam Height	Saponin
Shikakai	3 cm	Present
Seto Siris	3 cm	Present

Table 1 shows the results of the saponin test. The presence of foam after warming the solution indicates the presence of saponin.

Surface Tension Measurements :

The most common property of surfactants in aqueous solution is the reduction of surface tension. Surface tension reduction from 72 mN/m to 32–37 mN/m is considered to be good detergency [30]. Fig. 3 represents our preliminary results on surface tension of surfactant solutions. Fig. 3(a) shows surface tension as a function of concentration.The surface tension decreases with increase in concentration. This is due to breaking of hydrogen bonds when surfactants are added in water, leading to higher adsorption at the air-water interface. This allows an increase in interfacial area without overall increase in energy [31].

As can be seen in Fig. 3(a), the surface tension of Shikakai solution reduced to 35.6 $\pm$ 0.2 mN/m at a concentration of 1.0 $\times$ 10^{-2} g/cc, while that of Seto Siris reduced to 46.6 $\pm$ 0.2 mN/m at 6.0 $\times$ 10^{-3} g/cc. Johnson's Baby Shampoo solution showed a surface tension of 30.9 $\pm$ 0.2 mN/m at 1.0 $\times$ 10^{-3} g/cc. This reduced surface tension allows the surfactant solution to penetrate and wet the hair [30].

The surface tension reduction by Shikakai indicates that it has the potential to possess good detergency [31].

The variation of surface tension of Shikakai (1.26 $\times$ 10^{-3} g/cc) and Seto Siris (7.9 $\times$ 10^{-4} g/cc) with temperature, measured from 25°C to 50°C is shown in Fig. 3(b). With increase in temperature the surface tension decreased steadily. This may

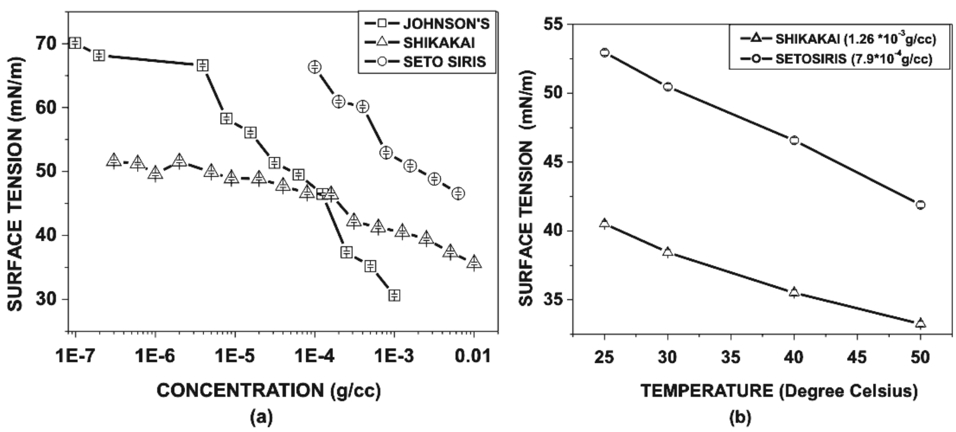

Fig. 3. (a) Variation of surface tension with concentration for different surfactants (at 25 ± 2°C). (b) Effect of temperature on surface tension of natural detergents.

be due to increase in the kinetic energy of the molecules which results in a decrease in the attractive forces between them [32].

Foaming Ability and Stability Studies :

The foaming abilities measured by the two methods are shown in Fig. 4. Foaming ability also increases with surfactant concentration. The qualitative behavior is same for both methods. However, the actual foam heights are much larger in the case of Bikerman's Method. This is due to the involvement of a large amount of gas compared to the Shaking Tube Method. The increase in foaming ability with concentration is due to large adsorption at the air-water interface leading to more surfactant in the foam film to stabilize the foam [33]. Foaming ability is highest for Shikakai followed by Johnson's Baby Shampoo and Seto Siris. The high foaming abilities of Johnson's Baby Shampoo and Shikakai may be due to dynamic reduction of surface tension and generation of large surface area required for foaming [24, 34].

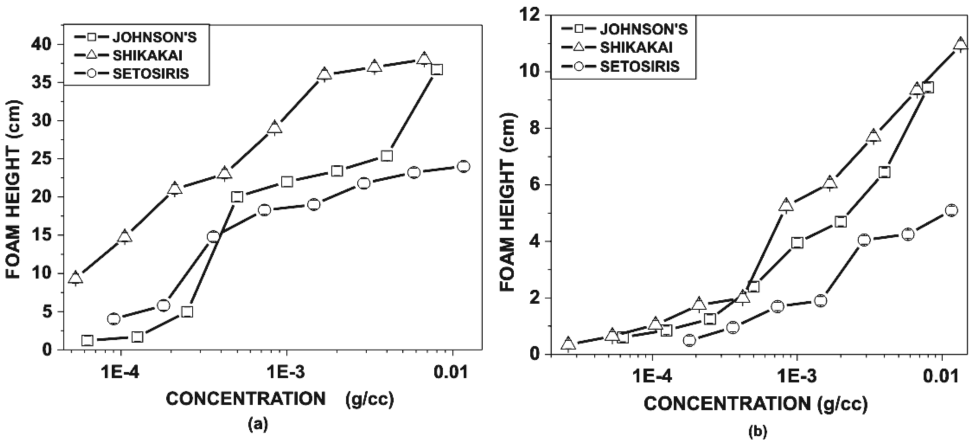

Fig. 4. Variation of Foam Height with concentration by (a) Bikerman's Method. (b) Shaking Tube Method.

In case of Johnson's Baby Shampoo a steep jump in foaming is seen for Bikerman's method when concentration increases from 2.5×10^{-4} g/cc to 5×10^{-4} g/cc. A large change is seen in surface tension (46.46 ± 0.2 to 35.19 ± 0.2 mN/m) when the concentration changes from 1.25×10^{-4} g/cc to 5×10^{-4} g/cc. This change in surface tension might have made it possible to increase the surface area easily [35]. While there is an increase in foaming by the Shaking Tube Method, it is not so sharp. Surface elasticity studies need to be carried out to understand this phenomenon better.

Production of foam by mechanical agitation results in an unsteady thermodynamic system. When foam is set to rest, it decays; the decay rate defines foam stability [36]. The decay of foam height against time are shown in Figs. 5(a),(b),(c) by Bikerman's Method and 5(d),(e),(f) by Shaking Tube Method. All the surfactants show an initial decrease of foam height, followed by decrease at a slightly reduced rate.

For Johnson's Baby Shampoo, foam height decreases exponentially for all concentrations. The foam totally dies out at lower concentrations for Bikerman's Method (Fig. 5a) while the foam is very steady even for low concentrations by Shaking Tube Method (Fig. 5d).

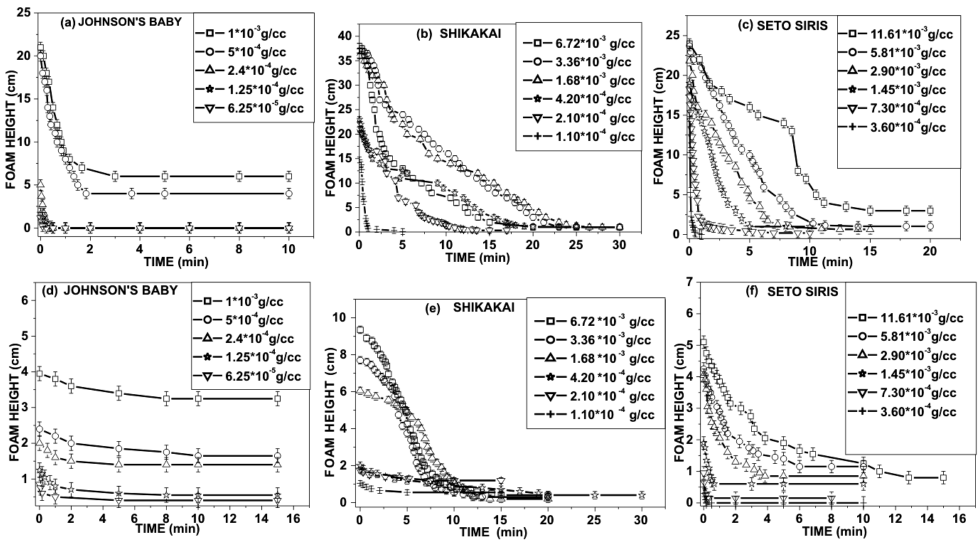

Fig. 5. Foam height against time by (a), (b), (c) Bikerman's Method and (d), (e), (f) by Shaking Tube Method.

The decrease is not always exponential for the natural detergents. In natural detergent solutions, many surfactants are probably present, which dominate at different time scales and concentrations, leading to complicated decay patterns. More experiments need to be performed to understand this decay pattern. In Johnson's Baby Shampoo, probably a single surfactant is dominant, producing a simple exponential decay. Also, a large number of oxygen atoms are present in natural detergents, leading to intermolecular and intra-molecular hydrogen bonding. This leads to decrease in the number of surfactant molecules reaching the air water interface, leading to lower foam stability. The foam stability decreases with decreasing concentration due to lower amount of surfactant at the air-water interface [32]. The stability of Seto Siris foam is comparable to Johnson's Baby Shampoo. Shikakai shows an unusual behavior, the stability initially increases with concentration, and then decreases after about 1.0×10^{-3} g/cc. This behavior requires further characterization.

The R5 value of 50% is taken as a cut off between metastable foams and foams of lower stability [22]. Fig. 6 show R5 values as a function of concentration for the two methods. A sharp transition can be seen from a region of low stability to high stability, the cut off concentrations given in Table 2. The transition is not observed by Shaking Tube Method for Seto Siris, probably due to the less amount of gas involved leading to low foaming. Alternatively, the quantity of surfactant present may not be sufficient. It is necessary to investigate at higher concentrations.

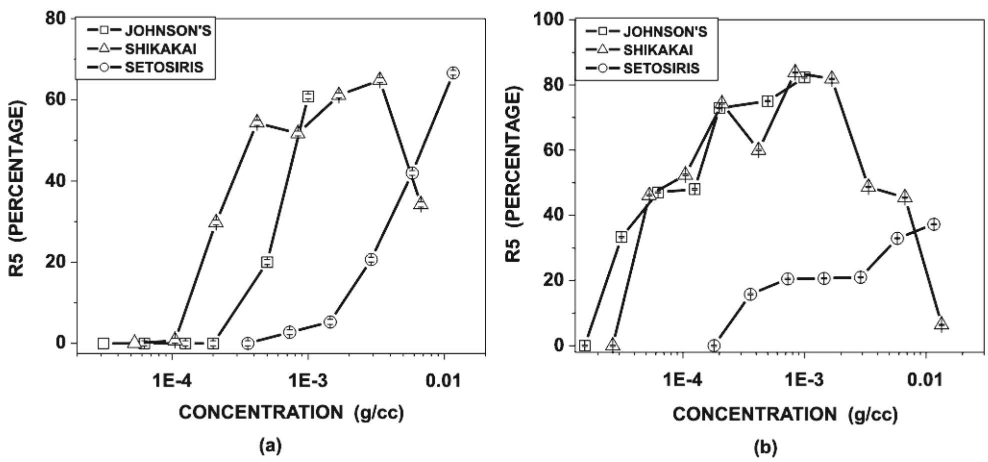

Fig. 6. R5 value against concentration by (a) Bikerman's Method (b) Shaking Tube Method.

TABLE 2.

Cut off Concentration from Low Stability to Metastable foam

Surfactants	C_1 (Bikerman's)	C_1 (Shaking Tube)
Johnson's Baby Shampoo	1×10^{-3} g/cc	1×10^{-4} g/cc
Shikakai	3×10^{-4} g/cc	3×10^{-5} g/cc
Seto Siris	5×10^{-3} g/cc	$>1 \times 10^{-2}$ g/cc

In Shikakai, the R5 value increases and then decreases with concentration. This phenomenon is seen for both methods and is not mentioned in the literature for any surfactant. Further work is needed to understand this.

Factors Affecting Foaming Properties of Natural Detergent :

Soaking time :

In order to see the effect of soaking time on foaming, the samples were soaked for 24 hours. Fig. 7 show foaming properties for samples soaked for 24 hours. Foam height is generally higher, probably due to increased surfactant concentration. However, the qualitative behavior remains similar.

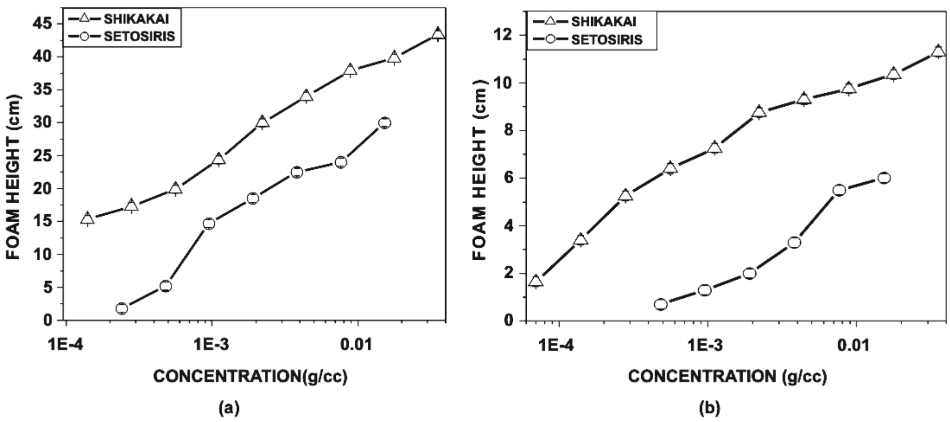

Fig. 7. Effect of 24 hrs soaking (a) Bikerman's Method (b) Shaking Tube Method.

Temperature :

The variation in saponin extraction due to temperature was studied by soaking the samples for an hour, followed by heating to temperatures up to 60°C. The results are shown in Fig. 8. The foam height first increased and then decreased. The increase would indicate better surfactant extraction at higher temperatures. The consequent decrease could be due to surfactant degradation at higher temperatures. This indicates that there is an optimum temperature for best saponin extraction.

Comparison of Surfactants :

Comparison of the foaming ability and stability of the surfactants is shown in Table 3. The Table show the concentration required for each surfactant for a foam height of 2 cm and R5 value of 50%. The trends are similar for both methods. Shikakai

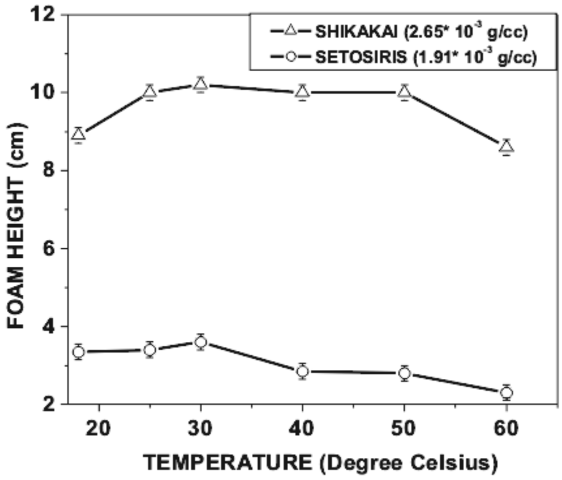

Fig. 8. Effect of temperature on foam height.

TABLE 3.

Comparison of concentrations for equal foaming ability and stability of different surfactants. All concentrations are in g/cc.

Surfactants	Foam height of 2 cm		R5 value of 50 %	
	Shaking Tube	Bikerman's Method	Shaking Tube	Bikerman's Method
Johnson's Baby Shampoo	$(3\pm0.1)\times10^{-4}$	$(4\pm0.5)\times10^{-5}$	$(1.75\pm0.1)\times10^{-4}$	$(8\pm0.6)\times10^{-4}$
Seto Siris	$(1\pm0.1)\times10^{-3}$	$(4\pm0.5)\times10^{-4}$	$> 11\times10^{-3}$	$(7.5\pm0.6)\times10^{-3}$
Shikakai	$(2\pm0.1)\times10^{-4}$	$(2\pm0.5)\times10^{-5}$	$(8\pm0.1)\times10^{-5}$	$(4\pm0.6)\times10^{-4}$

required a lower concentration, comparable to Johnson's Baby Shampoo. This shows that Shikakai has good foaming properties. Seto Siris required a much higher concentration to show the same foaming ability and stability.

In case of Bikerman's method, a lower concentration provides a 2 cm foam height than that providing 50% R5 value. The opposite is true for the Shaking Tube Method. This could be due to the mechanism of foam formation in the two methods.

When a lot of air is introduced into the liquid suddenly in Bikerman's method, foam is formed but it is not stable as not enough surfactants are present at the interfaces. In the Shaking Tube Method, a small amount of air is introduced, hence less foam is formed. Also, there is vigorous shaking which moves the molecules all around. Hence there are more surfactant molecules present at the interfaces, making the foam more stable.†

Wetting Ability :

Fig. 9 shows the wetting ability of the surfactants. As seen Shikakai and Johnson's Baby Shampoo showed better wetting ability than Seto Siris.

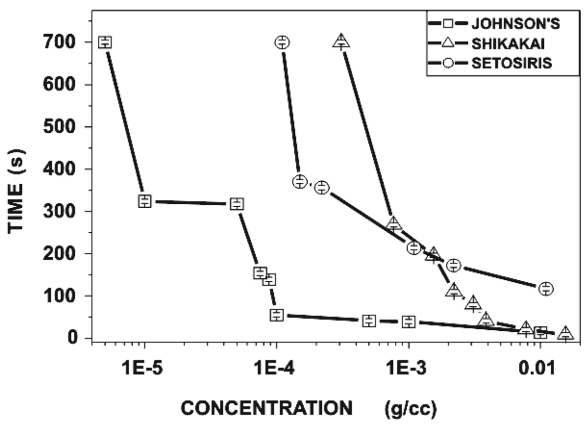

Fig. 9. Variation of wetting time versus concentration of surfactants.

Dirt Dispersion :

For Shikakai and Seto Siris, moderate amount of ink was seen in the foam, as shown in Fig. 10. The bottles on the left contain only surfactant solution while the one on right has surfactant solution with India ink. For Johnson's Baby Shampoo, the amount was light at higher concentrations and moderate at lower concentrations. This shows that Shikakai and Seto Siris can clean but not as good as Johnson's Baby Shampoo.

Cleaning Ability :

The percentage of cleaning as a function of concentration is shown in Fig. 11. The percentage of cleaning increases with concentration. The amount of dirt removed is different for different shampoos. Preliminary cleaning test shows that Shikakai's cleaning ability is comparable to Johnson's Baby Shampoo. Seto Siris has a somewhat lower cleaning ability.

†We thank one referee for attracting our attention to this fact.

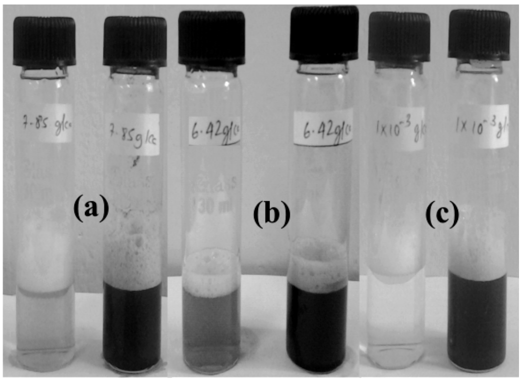

Fig. 10. Dirt dispersion of (a) Shikakai (7.85×10^{-3} g/cc) (b) Seto Siris (6.42×10^{-3} g/cc) and (c) Johnson's Baby Shampoo (1×10^{-3} g/cc). As seen, the foam in (c) has lesser India ink than (a) and (b).

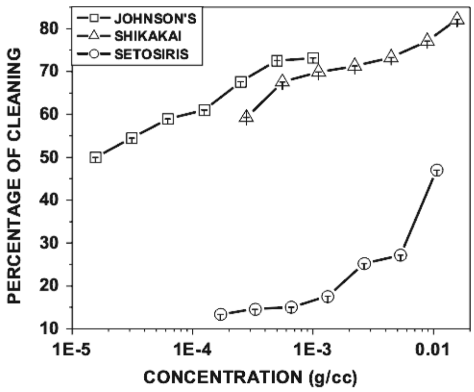

Fig. 11. Percentage of cleaning with concentration of the surfactants.

CONCLUSION

Our paper compares the properties of two plant extracts with a commercially available hair care product. We see that Shikakai is a potential candidate for hair care due to its large reduction of surface tension, good foaming properties, wetting and cleaning ability. It is a well-known material in hair care. There are numerous works on Shikakai [37] and the extracted saponin has been studied for many purposes.

However, very few workers have studied Shikakai systematically for cleaning purposes.

Seto Siris is more modest in its properties. However, since it is widely available, large quantities can be used. Our literature studies show that the foaming and cleaning properties of Seto Siris have been studied for probably the first time here. It has been traditionally used for hair care in the eastern Himalayas; a scientific study is required to quantify the properties of the plant.

The experiments with 24 hours soaking suggest that foaming in general improves with increasing soaking time. The natural surfactants also show an optimum temperature for surfactant extraction.

We have compared the foam produced using Bikerman's method and Shaking Tube Method. We find that whereas Bikerman's method produces more foam, Shaking Tube Method produces more stable foam. This effect is most remarkable for Johnson's Baby Shampoo.

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