



## STUDIES ON THE EMULSIFYING PROPERTIES OF GUM RESIN OF SHOREA ROBUSTA

N.K. SARATHCHANDRAPRAKASH\*<sup>1</sup>, M.VIJAYAKUMAR<sup>1</sup>, K. MANRAL<sup>1</sup>,  
U.V.BABU<sup>1</sup> AND D.VISHAKANTE. GOWDA<sup>2</sup>

<sup>1</sup>Research and Development Department, The Himalaya Drug Company, Bangalore, India

<sup>2</sup>Professor, Department of Pharmaceutics, JSS College of Pharmacy, JSS University, Mysore, India.

### ABSTRACT

In recent years, the importance of natural emulsifiers has increased considerably due to the safety concerns associated with synthetic emulsifiers. In this study, gum resin of *Shorea robusta* was evaluated for its emulsifying property. An emulsion was prepared using *S.robustasemi*-purified gum resin (HGRS) as an emulsifier (2.0, 3.0 and 4.0% w/w), and a standard emulsion was prepared with the known emulsifier, Glycerol monostearate (GMS SE). The prepared emulsions were evaluated for rheological properties, especially flow behavior, and they were stored at room temperature for stability studies at various time intervals for three months. Results of the study suggested that the semi-purified gum resin of *S.robusta* possesses good emulsifying properties. The emulsion formulated from the natural emulsifier was less sensitive to centrifugation, pH and shear stress. Thus, gum resin of *S.robusta* can be used as a natural emulsifying agent in topical preparations.

**KEY WORDS:** *Shorea robusta*, Gum resin, Natural emulsifier, Rheology

\*Corresponding author



**N.K. SARATHCHANDRAPRAKASH**  
Research and Development Department, The Himalaya Drug Company, Bangalore, India

## INTRODUCTION

A majority of personal care products and topical formulations are emulsions. The key ingredients of these formulations are emulsifiers, emollients and consistency enhancers. An emulsion is a dispersion of droplets of immiscible liquid in a continuous phase. An emulsifier is a substance that stabilizes an emulsion by different chemical and physical mechanisms, such as surface tension, repulsion and viscosity modification.<sup>[1]</sup> Synthetic emulsifiers are widely used in topical applications such as Betaine, carbomer, carboxymethyl cellulose, cetaryl alcohol, coco-betaine, ethyl acetate, glyceryl dioleate, PEG family, Sorbitan stearate, etc. Synthetic emulsifiers are known mild skin and eye irritants, and react with nitrites to form the highly potent carcinogenic nitrosamines. Due to the safety concerns associated with synthetic emulsifiers, there is a considerable increase in the importance of natural emulsifiers. But there are only a few known effective natural emulsifiers, making it difficult for formulators to avoid using synthetic emulsifiers completely. Some of the natural emulsifiers already in use are lecithin, gum acacia and gum tragacanth.<sup>[2,3]</sup> *Shorea robusta* Gaertn.f. is a species of tree belonging to the Dipterocarpaceae family and is widely distributed in the Indian subcontinent, Indonesia, Malaysia and Philippines. Resin obtained from the plant is traditionally used to treat viral skin ailments and reported to possess wound healing activities.<sup>[4]</sup> Gum resin of *S. robusta* contains triterpenoids, ursolic acid, amyrenone, and b-amyryn along with polyphenols and carbohydrates.<sup>[5]</sup> The present study is an attempt to evaluate the emulsifying property of the gum resin of *S. robusta*.

## MATERIALS AND METHODS

### Chemicals

The gum resin of *S. robusta* used in this study was procured from the local market. The sample specimen was authenticated by Dr. Kannan, Botanist, and a voucher specimen was deposited in the herbarium at The Himalaya Drug Company, Bangalore. Chemicals procured for preparing emulsions were light liquid paraffin IP grade and Glyceryl

monostearate, Glycerin IP grade, which was used as synthetic self-emulsifier (GMS SE). All the other chemicals were of analytical grade.

### Preparation of Gum Resin Fraction

Dried gum resin of *S. robusta* was powdered and extracted with hexane. Filtered extract was concentrated under reduced pressure using a Buchi Rotavapor. The yield obtained was 10%w/w (HGRS). The dried hexane extract was used as emulsifier and studied for its emulsifying property.

### Formulation of Emulsion

Emulsion was prepared using *S. robusta* semi-purified gum (HGRS) as an emulsifier with light liquid paraffin, glycerin and water. The gum concentration levels used in the test emulsion were 2.0, 3.0 and 4.0% w/w. Standard emulsion was prepared with a known emulsifier, Glyceryl monostearate (GMS). In both the emulsions, phenoxyethanol was used as a preservative. Thus, prepared formulations were tested for emulsion characterization and stability studies.

### Characterization of Emulsion

#### Dye Test for Identification of Emulsion

In this test, an emulsion is mixed with a water-soluble dye amaranth and a drop of it is placed on a slide and observed under microscope. If the continuous phase appears red, the emulsion is oil/water (o/w) type, as water is in the external phase and the dye dissolves in it to give the red color. If the scattered globules appear red and continuous phase appears colorless, the emulsion is Oil/water (O/W) type.

### Rheological Evaluation

The prepared emulsions were subjected to rheological parameter evaluations, especially flow behavior by using a rheometer (Model 302, Anton-Paar). The emulsions were dependent on the geometry, and according to the applied shear rate viscosity was checked. Flow curve was obtained by using the oscillating mode (amplitude sweep). Experiments were conducted at 25°C by controlling the temperature using the Peltier system. The geometry used was parallel plate

(PP50). A shearing force was applied to the top of the rectangle while the bottom was held in place. The resulting shear stress,  $\tau$ , deforms the rectangle into a parallelogram. The area involved would be the top of the parallelogram. The shear stress was applied equally to all the four formulations. Rheology profiles were observed for both the modules - Storage module ( $G'$ ) and Loss module ( $G''$ ). All results are expressed in graphical presentation, and the rheology profile of the natural emulsifier was comparable with the synthetic emulsifier.

#### **Determination of globule size**

All the emulsions were examined for their globule<sup>[7]</sup> size and structure by optical microscopy. A drop of homogenous emulsion was placed between microscope slide and coverslip. The emulsion structure was then observed using a conventional optical microscope. Mean of the globule size ( $X$ ) was calculated as  $X = \sum fx / \sum f$  (Where 'f' is the frequency of each size. For each emulsion sample, 3 representative).

#### **Stability Study of Emulsion**

##### **Estimation of creaming power**

In a 100 ml graduated measuring stopper cylinder, the emulsion was transferred, filled up to the mark and stored at room temperature ( $25 \pm 2^\circ\text{C}$ ) for three months. Cream is the separation of emulsion into opaque layer at the top and turbid layer at the bottom. The extent of creaming was estimated by percent creaming =  $\text{HC}/\text{HE} \times 100$ , where, HC is the height of the cream layer and HE is the height of the original emulsion.<sup>[6]</sup>

##### **Centrifuge test**

All the emulsions were filled in 15 ml capacity centrifuge test tubes and placed in the centrifugal apparatus (Remi Centrifuge apparatus). The centrifuge was set for 60 minutes at 3000 rpm. After the completion of the set program, test tubes were taken out and observed for any phase separation.

##### **Emulsion stability with pH variations**

Variation in pH has an effect on the emulsion stability. All the emulsions were prepared at varying pH points of 4.0, 7.0 and 9.0 using glacial acetic acid and Triethanolamine, and their emulsion stability observed.

#### **Emulsion stability with thermal effect**

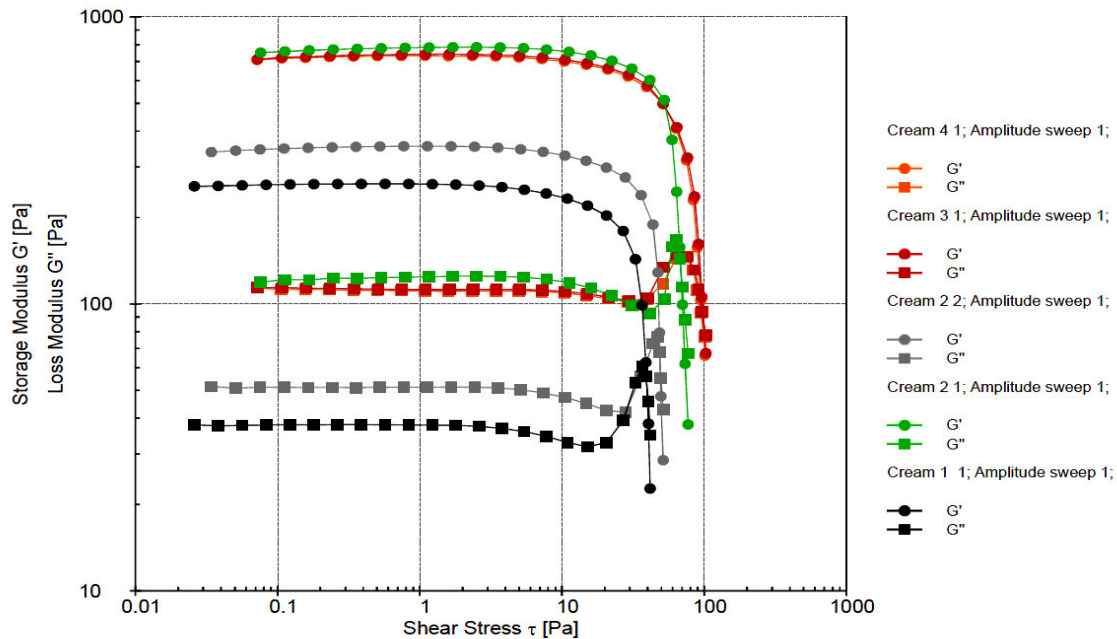
The emulsifier strength varies with difference in temperature. The emulsions were prepared and stored at different temperatures of 4, 25 and  $50 \pm 2^\circ\text{C}$  for 12 weeks. Emulsion stability was evaluated by subjecting the samples to centrifugation at regular intervals and checking for phase separation.

## **RESULTS AND DISCUSSION**

In the present study, the emulsifying properties of the hexane fraction of gum resin of *S. robusta* (HGRS) were evaluated. Several tests were conducted to assess the characterization and stability of the emulsion. Emulsions were prepared with hexane fraction of gum resin of *Shorea robusta* as a natural emulsifier and concentrations used were 2.0, 3.0 and 4.0 %w/v. Concentrations were arrived based on preliminary experiments on evaluation of the emulsifying properties of *S. robusta* gum resin. Prepared formulations were tested for emulsion characterization and its stability with compared to standard emulsion containing known emulsifier Glyceryl mono stearate<sup>[9]</sup>. Results of the dye test showed that the dye was distributed as colored continuous phase and the droplets were colorless. The oil forms the dispersed phase and water forms the continuous phase, and it was the w/o type of emulsion. Emulsion stability can be assessed by rheological parameters<sup>[8]</sup> using rheometer by two techniques: Long-term storage stability can be assessed i.e. the behavior in a state of rest following a long period of time in the state of rest. using oscillatory tests such as an amplitude sweep or a frequency sweep. The stability directly after vigorous shearing can also be observed using structural regeneration in the form of step tests. In both the options, the sample is judged at rest, though the previous conditions may differ. Oscillatory tests are best used for the analysis. The measuring bob oscillates with a defined deflection (amplitude) and frequency around its rotational axis. The sample's resistance due to the shearing is then recorded by the rheometer measuring head as two independent raw values (torque and time phase shift). These two values are then presented as the storage modulus  $G'$  and the loss modulus  $G''$  using a software program.  $G'$

describes the elastic part and  $G''$  the viscous part of the viscoelastic behavior. Rheology profile of the prepared emulsion was observed for both Storage module ( $G'$ ) and Loss module

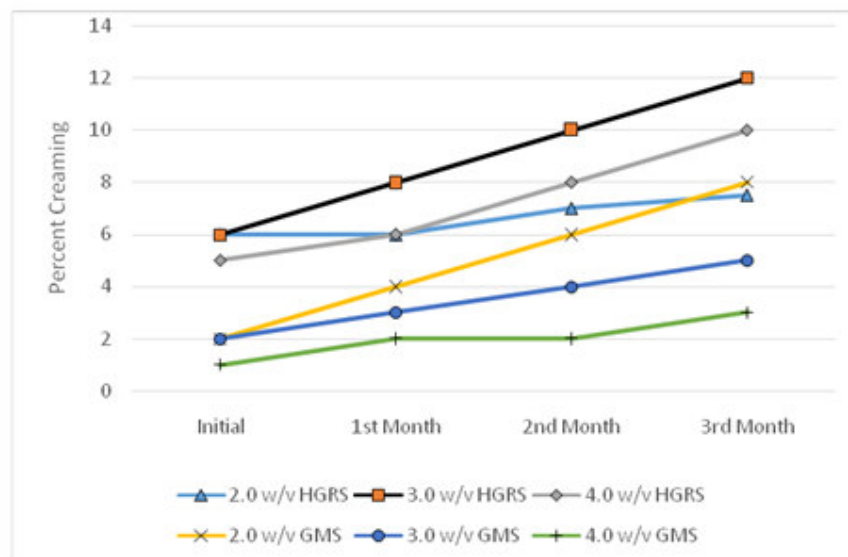
( $G''$ ). All the results are expressed in graphical presentation. The rheology profile of the natural emulsifier was comparable with the synthetic emulsifier (Figure 1).



**Figure 1**  
**Amplitude Sweep Cream-1 (2% GMS); Cream - 2 (4% GMS);**  
**Cream - 3 (2% HGRS); Cream - 4 (4% HGRS)**

The process by which an emulsion completely breaks into bulk oil and water phases is generally considered to be governed by four different droplet loss mechanisms, i.e., Brownian flocculation, creaming, sedimentation flocculation and disproportionation. Creaming is the process by which buoyant emulsion droplets tend to rise to the top of a container. Figure 2 shows the creaming behavior of the emulsions prepared both with HGRS and GMS; it illustrates that the emulsion prepared with HGRS is more stable when compared with the emulsion prepared with GMS. The difference in their creaming rates may be attributed to the variation in their emulsifying properties—lower the emulsifying property, larger the oil

globules and thereby greater the rate of creaming. The kinetics of the emulsion-breaking process is governed by three different particle loss mechanisms: Brownian flocculation, sedimentation flocculation and creaming. Globules under Brownian motion collide and then coalesce to form larger globules. At the same time, creaming in emulsions is also dependent on the difference in densities between the globules and continuous medium. Sedimentation flocculation occurs due to difference in creaming rates of globules of different sizes. Thus, larger globules cream out at a faster rate than the smaller ones, and therefore, larger globules collide with slower moving smaller particles as they cream out<sup>[7]</sup>.



**Figure 2**  
***Creaming behavior of emulsions prepared with HGRS and GMS***

Centrifugation tests were performed on both the emulsions immediately after their preparations. Emulsions prepared with HGRS did not show any phase separation after centrifugation at 3000 rpm for 60 minutes; this proved that the emulsions prepared with HGRS were stable. The effect of pH was observed on the emulsions prepared with both

HGRS and GMS. The emulsions prepared at different pH values did not show any phase separation and did not affect the globule size where globule size is not varied much against the regular intervals (Table 1). The thermal study proven that HGRS have ability produce the stable emulsion even in the stress conditions like 5°C, 25°C and even 50°C.

**Table 1**  
***Effect of pH on emulsions prepared with HGRS and GMS***

Concentration	pH	Average globule size (µm)			
		Initial	1 <sup>st</sup> month	2 <sup>nd</sup> month	3 <sup>rd</sup> month
2.0 w/v HGRS	4.0	46	48	53	60
	7.0	32	35	38	50
	9.0	50	56	62	66
4.0 w/v HGRS	4.0	42	50	55	60
	7.0	30	32	38	42
	9.0	53	58	61	69
2.0 w/v GMS	4.0	18	22	25	30
	7.0	11	15	17	22
	9.0	20	22	30	33
4.0 w/v GMS	4.0	15	18	22	25
	7.0	10	14	18	21
	9.0	12	15	18	25

## CONCLUSION

The results of the study showed that the semi-purified gum resin of *S.robusta* possesses good emulsifying properties. The emulsion prepared with natural emulsifier was less sensitive to centrifugation, pH and shear stress. Thus, gum resin of *S.robusta* can be used as a natural emulsifying agent in topical preparations. However, further detailed studies are needed to find out the emulsifying mechanism and the chemical nature of the gum resin.

## ACKNOWLEDGMENT

We would like to thank The Himalaya Drug Company management for all the help and support in conducting this experiment.

## REFERENCES

1. Tadros TF. Emulsion science and technology: A general introduction. In: Tadros TF, editor. Emulsion science and technology. Weinheim, Germany: Wiley-VCH Verlag GmbH & Co. KGaA; 2009. p. 1-56.
2. Choudhary PD, Pawar HA. Recently investigated natural gums and mucilages as pharmaceutical excipients: An overview. J Pharmaceut. Article ID 204849, (2014)
3. Sjostrom B, Bergenstahl B. Preparation of submicron drug particles in lecithin-stabilized o/w emulsions: I. Model studies of the precipitation of cholesteryl acetate. Int J Pharma, 84:107-16, (1992)
4. Wani TA, Chandrashekara HH, Kumar D, Prasad R, Gopal A, Sardar KK, Tandan SK, et al. Wound healing activity of ethanolic extract of *Shorea robusta* Gaertn. f. resin. Indian J Exp Biol. 50:277-81, (2012)
5. Rasheed NMA, Nagaiah K, Waheed MA. Phytochemical standardization of oleo resin of *Shorea robusta* Gaertn (Dipterocarpaceae) with modern analytical technique. Int J Phytomed, 4:503-10, (2012)
6. Panda DS. Studies on gum of *Moringa oleifera* for its emulsifying properties. J Pharm Bio Allied Sci, 6:92-6, (2001)
7. Reddy SR, Fogler HS. Emulsion stability: Delineation of different particle loss mechanisms. J Colloid Interface Sci, 79: 105-13, (1981)
8. Gallegos C, Franco JM, Partal P. Rheology of food dispersions. Rheology Reviews, 19-65. (2004)
9. Glyceryl Mono stearate: "Cosmetic info" Accessed on 13 November 2014. <http://www.cosmeticsinfo.org/ingredient/glyceryl-stearate>