
Rosin-based Polymers in the Preparation of Lotions

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In this study, rosin-based polymers were employed for the preparation of lotions. Rosin-based polymer was prepared and screened for its ability to be used as an emulsifying agent for the preparation of lotion. The lotion was formulated employing a blend of polymer 2 and Span 80. Another lotion was prepared with blend of Tween 80 and Span 80 for the purpose of comparison. The formulation was evaluated for visual appearance, viscosity and globule size under accelerated conditions. Lotions containing blend of polymer 2 showed good stability than lotion prepared with blend of Tween 80.

Lotions are liquid or semi liquid solutions, suspensions or emulsions intended for application without friction to the broken skin. The advantage of a lotion preparation is that it leaves a thin coating of medicament on the skin surface after solvent evaporation. Various surfactants and viscosity modifiers that are used in lotions include methylcellulose, carmellose sodium and sodium lauryl sulphate¹. Various natural agents are used as surfactants or viscosity modifiers in the preparation of lotions.

The stability of lotions depends to a large extent on the selection of suitable hydrophilic-lyphophylic balance (HLB) blend and type of surfactant used. The selection of a proper blend of surfactants often stands as a challenge to industrial scientists and research workers since many problems arise in the design and manufacture of pharmaceutical dispersions. Rosin-based polymers were preferred for the present study as they were expected to be biomedically safe because they are basically derived from rosin, which is a vegetable product widely used in food and confectionary industry².

Rosin is a solid resinous material obtained naturally

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from various varieties of pine tree. The principle commercial sources of rosin are *Pinus soxburghi*, *Pinus longifolium*, *Pinus palustris* and *Pinus toeda*. Rosin is composed of approximately 90% rosin acids and 10% non-acidic materials. Rosin acids are monocarboxylic acids having typical molecular formula $C_{20}H_{30}O_2$. There are two types of rosin acids; abietic acid type and pimaric acid type. Rosin is reported to have emulsifying property³. The use of rosin-based esters was previously reported as cream bases⁴. Rosin-based polymers are also reported to be useful for the preparation of stable w/o/w multiple emulsions⁵.

Based on the above documented information, series of rosin-based polymers (ten) with various compositions were prepared and screened for possible utility as surfactants in pharmaceutical preparations. These polymers were given numbers that correspond to the number of rosin derivatived used from polymer 1 to polymer 10. Amongst these polymers only polymer 2 is reported in the present study as it produced more stable lotions under accelerated conditions, compared to the other polymers tested.

MATERIALS AND METHODS

The materials used along with their sources are as follows: Rosin N grade (M/s Tayebai Ebrahimji Pettodwala, Mumbai), glycerol, sorbitol, pentaerythritol, maleic anhydride, pthaleic anhydride (Qualigen), castor oil (Samar

Chemicals, Nagpur) and potassium dihydrogen phosphate (SD Fine Chemicals, Mumbai). The polymers based on rosin are prepared in the laboratory.

Preparation, composition and physicochemical characteristics of rosin-based polymer 2:

Polymer 2 was prepared using the following process. Rosin 50 g, castor oil 35 g, glycerol 2.2 g, sorbitol 14.7 g, maleic anhydride 6 g and phthalic anhydride 2 g were placed in four-neck two-liter glass reactor. This reactor was fitted with a condenser, stirrer and temperature control arrangement. The reaction temperature was maintained within $\pm 2^\circ$ with the help of an accurate thermometer. At first rosin, part of maleic anhydride, castor oil and sodium sulphate and sodium bisulphate (catalysts) were added in the reactor and temperature was slowly raised to 160° . The reaction was maintained at this temperature for 1 h. After this the temperature was lowered to 120° and glycerol and sorbitol were added slowly in about 15 min. The heating was continued for 3 to 4 h at 210° to 250° . The reaction mixture was then cooled to 80° . At this temperature xylene (solvent), maleic anhydride and phthalic anhydride were slowly added in about 15 min. Further heating was done at lower temperature of 180° - 190° for 3 to 4 h till the desired acid value of 0.44 is reached. Finally xylene was stripped off totally by heating at 150° under vacuum. The sample was finally strained through a fine mesh and stored carefully. During the process acid value of the product was determined intermittently as reported previously⁶. The prepared polymer 2 has acid value 0.44, saponification value 107.35, Hydrophilic-lipophilic balance 12.7, viscosity 72000 centipoises, weight per ml 1.5389 and refractive index (1g solution) 1.3335.

Preparation of lotions:

Lotions were prepared using liquid paraffin 35 g, cetyl alcohol 1 g, wool fat 1 g, surfactant blend (polymer 2 and span 80 or tween 80 and span 80) 7 g and water upto 100 ml. Total percentage of oil phase in the above formulae is 37%. The required HLB for liquid paraffin, wool fat and cetyl alcohol is 12, 10 and 15 respectively. Therefore the total required HLB for 37% oil phase comes to 12.1 based on the contribution of each oil ingredient towards HLB. Span 80 is the common emulsifying agent and it has HLB value of 4.3. The HLB values of Tween 80 and polymer 2 are 15 and 12.7, respectively. The rosin-based emulsifying agent and Tween 80 represent the hydrophilic emulsifier, whereas, span 80 represents the lipophilic emulsifier. Hence the emulgents are blended with span 80 to achieve required

HLB ratio for the preparation of lotion.

Therefore, the concentration of Tween 80 and Span 80 used to prepare lotion was 5.11 and 1.89 g, respectively. The concentration of polymer 2 and Span 80 used to prepare lotion was 6.5 and 0.5 g, respectively. All the ingredients were weighed and placed in the laboratory blender with Span 80. The polymeric 2 was dissolved in distilled water and this solution is then added in the blender. The mixture was then stirred upto 10 min at maximum speed. The product was then kept overnight before subjecting to further analysis to attain equilibrium.

Irritation test:

Test for irritation was performed by human repeat insult patch test⁷. The informed consent of the volunteers was taken before commencing the studies. The study involved five men and five female volunteers. The study involved only topical application of lotions. Ten human volunteers of different age group were selected and 1 ml of lotion was applied on 8 cm² areas near to the elbow and covered with cotton. Subjects were evaluated after being exposed to patch for 10 times for 20 d and observed for any irritation or reaction.

Stability studies:

The prepared lotions were distributed in a 500 ml glass bottles and stored at two different temperatures i.e. room temperature ($25 \pm 3^\circ$) and $40 \pm 1^\circ$ for 42 d. The parameters of stability assessment were: viscosity, globule size, pH, conductivity and relative dielectric constant. Viscosity was determined using Brookfield Synchro-Lectric viscometer (Model RVT, Serial 50672, 230 volts, Frequency 50, Brookfield Engineering Labs. Inc.). The globule size of lotions was determined using Malvern particle size analyzer (Mastersizer u+ Ver. 2.12, Serial Number 33085-08, Malvern Instruments Ltd.). The pH of 1 g% of the lotions was determined using a pH meter (Hanna instruments). The conductivity was determined using digital conductometer (Elico Pvt. Ltd., Model: CM-180).

The relative dielectric constant was determined using Universal Dielectrometer, (Type: OH-301, No. 457). The measurement of relative dielectric constant was carried out by reading the capacitance of a condenser containing the lotion. A probe capacitance cell, which could be attached to the dielectrometer through a coaxial plug, was used to measure the pF value (capacitance). About 30 ml of lotion was taken into suitably sized cylinder and the head of the probe cell was dipped into the solution. Care was exercised to

take all the readings at same temperature and at the same height. All measurements were done at a fixed frequency of 3 mc/S. The conductivity measuring cell lends itself to relative measurement of pF value for comparison. The recorded pF values were used as such without further reduction and were thus designated relative dielectric constant (relative DEC). The relative DEC for distilled water was found to be 54 whereas the value of DEC was 80. The lotions were allowed to equilibrate to room temperature before taking the readings.

RESULTS AND DISCUSSION

It was observed that the prepared lotions show good spread ability and homogeneity during the period of storage at room temperature ($25\pm 3^\circ$) and $40\pm 1^\circ$ for 42 d. Lotions did not induce any skin irritation. The results of changes in viscosity of lotions on exposure to various temperatures are presented in fig. 1. The viscosity was found to decrease with the rise in storage temperature for all the lotions tested. The viscosity of lotions prepared with blends of polymer 2 was seen to decrease from 86 to 56 centipoises on storage at 40° for 42 d. In case of lotions prepared with blends of Tween 80 there was decrease in viscosity from 49.5 to 29

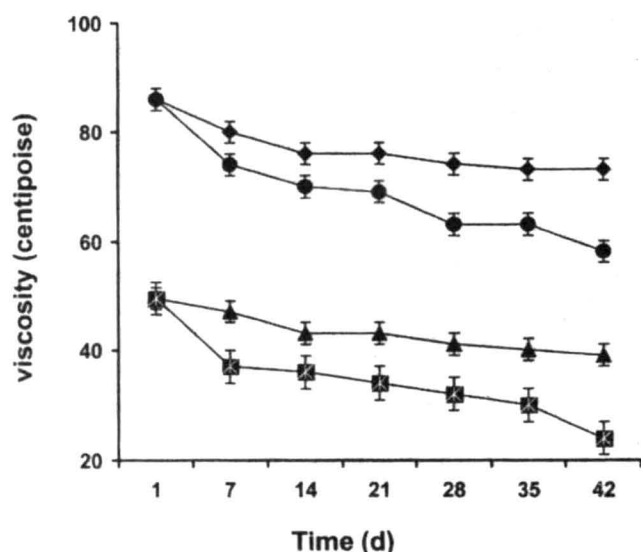


Fig. 1: Viscosity changes in lotions.

RL 2 is the lotion prepared with a blend of polymer 2 and Span 80 stored at room temperature (-♦-). RLT is the lotion prepared with a blend of Tween 80 and Span 80 stored at room temperature (-▲-). 40 L 2 is the lotion prepared with a blend of polymer 2 and Span 80 stored at 40° (-○-). 40 LT is the lotion prepared with a blend of Tween 80 and Span 80 stored at 40° (-■-).

centipoises when observed at 40° for 42 d. The correlation coefficient of viscosity for lotions prepared with blends of polymer 2, and Tween 80 was found to be 0.8977 and 0.948, respectively. It seems clear from above data that decrease in viscosity with respect to days is greater in case of lotions with blend of Tween 80 as compared to lotions with blend of polymer 2 and span 80.

The changes in globule size of the lotions during storage are presented in fig. 2. The globule size of lotions prepared with blends of polymer 2 increased from 2.12 to 2.62 μm on storage at 40° for 42 d. The increase in globule size from 1.35 to 2.36 μm was observed in case of lotions prepared with blends of Tween 80. The correlation coefficient of globule size for lotions prepared with blend of polymer 2 and Span 80 and blend of Tween 80 and Span 80 was found to be 0.9066 and 0.9495, respectively. It is clear from the above data that increase in globule size with respect to time is more for lotions prepared with blends of Tween 80 and Span 80 compared to the blend of polymer 2 and Span 80. The globule size of lotions prepared with polymer 2 was slightly affected at both the storage temperatures as compared to the lotions with Tween 80. The rise in particle size

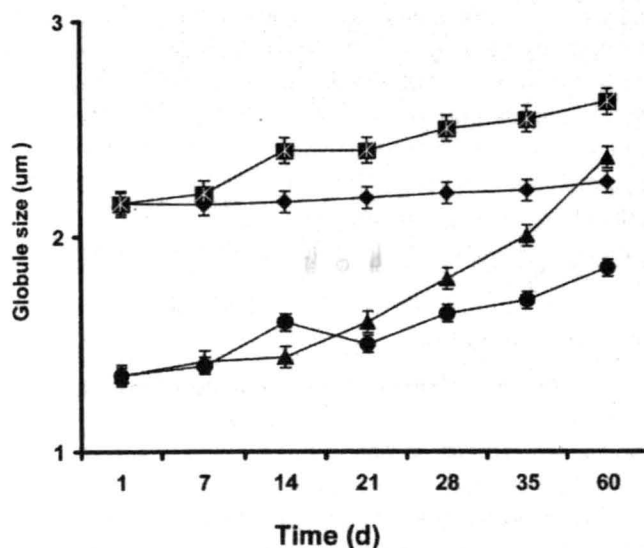


Fig. 2: Globule size changes in lotions.

RL 2 is the lotion prepared with a blend of polymer 2 and Span 80 stored at room temperature (-♦-). RLT is the lotion prepared with a blend of Tween 80 and Span 80 stored at room temperature (-▲-). 40 L 2 is the lotion prepared with a blend of polymer 2 and Span 80 stored at 40° (-○-). 40 LT is the lotion prepared with a blend of Tween 80 and Span 80 stored at 40° (-■-).

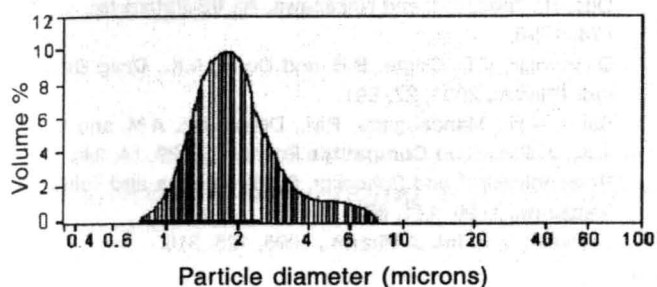


Fig. 3a: Globule size distribution in Polymer 2 lotions on Day 1.

Initial particle size distribution of globules in lotion prepared with a blend of polymer 2 and Span 80 on Day 1.

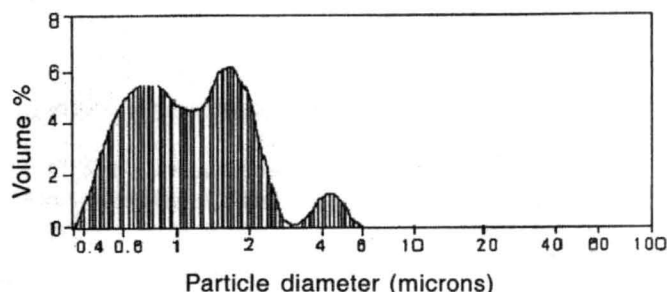


Fig. 3b: Globule size distribution in Tween 80 lotions on Day 1.

Initial particle size distribution of globules in lotion prepared with a blend of Tween 80 and Span 80 on Day 1.

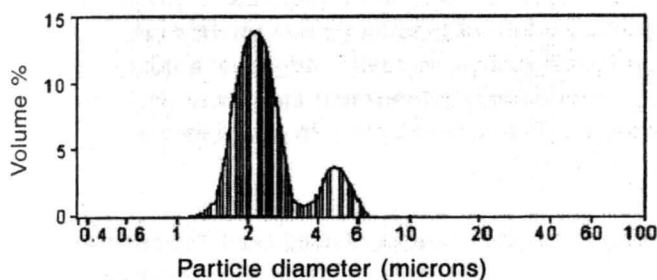


Fig. 4a: Globule size distribution in Polymer 2 lotions on Day 42.

Particle size distribution of globules in lotion prepared with a blend of polymer 2 and Span 80 after storage for 42 d.

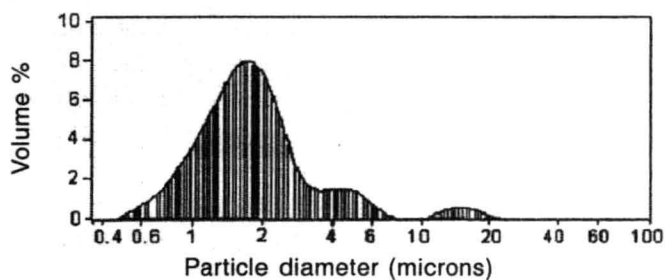


Fig. 4b: Globule size distribution in Tween 80 lotions on Day 42.

Particle size distribution of globules in lotion prepared with a blend of Tween 80 and Span 80 after storage for 42 d.

was found to be more in case of lotions prepared with Tween 80 and Span 80 and stored at 40°. The globule size data is in good agreement with viscosity measurements. Fig. 3(a, b) and fig. 4 (a, b) shows the details of the particle size analysis during 42 d of ageing for lotions stored at room temperature (25±3°) and 40±1°. As shown in figure there was a change in the particle size for lotions prepared with blend of polymer 2 and Tween 80 from 2.15 to 2.62 microns upon storage at 40° for 42 d. The particle size of lotions prepared with blend of Tween 80 and Span 80 was changed from 1.35 to 2.37 microns upon storage at 40° for 42 d.

It is well-established facts that increase in temperature affects the stability of the emulsions under investigation⁶. The rise in temperature affects the changes taking place at the interface including adsorption or desorption of surfactants from the interface leading to increase in globule size and subsequent decrease in viscosity. As the viscosity decreases the tendency of oil globules to come closure increases which further increases the globule size due

to agglomeration. It is worth to mention that as the chemical structure of the prepared rosin-based surfactant for the present studies is not known it would be difficult to precisely find out the changes taking place at the interface. However present study suggests the utility of such rosin-based polymeric surfactants to prepare stable lotions compared to the Tween 80 based surfactants.

It is concluded from the present study that lotions prepared using blend of polymer 2 and Span 80 show good storage stability than the lotions prepared with blend of Tween 80 and Span 80. The rosin-based polymer can be successfully used for the preparation of lotions with better storage stability.

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