Comparative studies of the effect of storage on the physiochemical properties of moisturising creams produced from Delonix regia seed oil and mineral oil

Storage conditions are very important in determining the quality of products as products are not used immediately after production, they have to pass through various storage processes before getting to the end user. The aim of this article is to study the effect of storage on moisturizing creams produced from Delonix regia seed oil (vegetable oil) and mineral oil. Oil in Delonix regia seeds was extracted using cold maceration and their physiochemical compositions were evaluated using AOAC 2016 methods while their elemental mineral components were analysed using Atomic Absorption Spectrophotometer (AAS). The oils from D. regia and Mineral oil were then used in the production of cosmetic cream products and their physiochemical properties were characterized. Specific gravity, viscosity, refractive index, acid value, peroxide value, iodine value, saponification value and unsaponifiable matter were: 0.99g/cm3, 0.87g/cm3; 47.60, 11.52; 1.4335, 1.5246; 22.44mgKOH/g; 7mgEq/kg; 37.56g/100g; 30mgKOH/g and 2.81, 1.02 respectively. Magnesium (7.56332ppm) was detected as the mineral with the highest concentration in D. regia oil cream while Na (9.4560ppm) was the mineral with the highest concentration in mineral oil cream. For D. regia seed oil cream, the pH, non-volatile matter, and moisture content increased as the storage period increased while the total fatty matter and specific gravity decreased as the storage period increased. For the mineral oil, pH, non-volatile matter and moisture content decreased as the storage period increased while the specific gravity and total fatty matter increased as the storage period increased. This study has indicated that the storage period has a significant effect on the physiochemical properties of moisturizing creams from both Delonix regia seed oil and mineral oil. However, the physiochemical properties are still within the acceptable limits and standard as set by the Standard Organisation of Nigeria (SON).

Keywords— Storage condition, Delonix regia oil, Mineral oil, Seed oil, Physiochemical properties, Cosmetic creams

1. INTRODUCTION

Cosmetic products have a life of their own and most ingredients used in cosmetics formulation have extended shelf life in their pure state. Emulsions are produced by mixing active ingredients, lipids, and oils with the help of emulsifiers and water.

Oil in water (O/W) or water in oil (W/O) emulsions are unstable, usually separating and splitting into two distinct phases. This instability can be manifested at different time rates and through a variety of physicochemical destabilizing processes (Akhtar et al., 2011). The interaction between the different substances will occur and components may react chemically with each other, separate physically or degrade due to their water content. As a matter of fact, consumers prefer the longest possible shelf life with the quality of the product retained.

The first production of mineral oil was not known, but as a derivative of petroleum, it must have been after the discovery of crude oil. Mineral oil is an effective skin moisturizer providing occlusivity and emolliency. Its occlusive effects lead to increases in stratum corneum water content by reducing trans-epidermal water loss. Mineral oil is used to treat dry skin conditions in various cosmetic products. It has been shown to improve skin softness better than wax esters, triglycerides, and fatty acids. Its effect is largely confined to the epidermal layers, and as a result of its limited penetration, it is considered to be a very safe ingredient for cosmetic use (Rawlings et al., 2012).

Delonix regia is a legume belonging to the family of Caesalpiniaceae. It is a semi-deciduous tree known as flame of the forest in Nigeria and grows to heights of about 18 meters. The fruits are long pods, which dangle from the branches, they are green when...
The seeds of Delonix regia yield thick and dark green oil for medicinal use, hair grease and a useful alternative source of biodiesel. Oils extracted from the plant sources have a rich history of use by local people as a source of food, energy, medicine and for cosmetics application. It has been used in the production of lubricants, soaps and personal body care products, as well as in the topical treatment of various conditions such as hair dandruff, muscle spasms, varicose veins, and wounds. (Edward et al., 2013).

Products with extended shelf life usually contain substances which are highly stable in chemical and microbiological respect. Therefore, there is a need to study the physicochemical properties of products within the period of storage. This work is aimed at studying and comparing the effects of storage period on the physiochemical properties of oil in water emulsion body creams made from Delonix regia seed oil and petroleum mineral oil.

2. MATERIALS AND METHODS

Delonix regia pods were collected from the parent trees at Ibadan, Oyo State, Nigeria. The D. Regia seeds were obtained by cracking the pod and the seeds were sorted to remove foreign materials, pulverized and milled for easy oil extraction. Mineral oil was purchased from a local chemical market in Ojota, Lagos State, Nigeria.

2.1 Extraction of oil

The oil extraction was carried out via cold maceration according to the method described by Ewansiha et al., (2012). The milled D. regia sample (1500g) was soaked with hexane (2000mL) in 2.5L amber Winchester bottles for 48 hours. The mixture was later filtered using a muslin cloth and cotton wool to separate the hexane-extracted D.regia seed oil from the seed cake. The D. regia oil was successfully recovered from the D.regia oil - hexane extraction mixture using a rotary evaporator (N-1001S-WD Oasis Scientific Inc.)

2.1.1 Physicochemical characterization of the D. regia oil and mineral oil: The physicochemical properties, such as oil yield, pH, specific gravity, refractive index, viscosity, acid value, peroxide value, iodine value, saponification value and unsaponifiable matter of D. regia oil were all determined using AOAC (2016) methods.

2.2 Formulation of water in oil moisturizing cream

The Delonix regia seed oil and mineral oil were used in the production of moisturising creams following the method prescribed by Oyedeji et al., (2010) with some modifications. The materials were grouped into aqueous and lipid phases. The lipid phase was accomplished by adding the D. regia oil in a stainless steel container and mixed with other fat-soluble materials such as emulsifying wax, cetyl alcohol, stearic acid, propyl paraben and vitamin E. The mixture was then heated to about 75°C. The aqueous phase was accomplished by adding water-soluble materials such as glycerol, sodium citrate and methyl paraben to the deionised water in a stainless container. The mixture was also heated to the same temperature as 75°C as the lipid phase. The aqueous phase was added slowly to the lipid phase and stirred vigorously with the help of homogenizer at 2000 revolutions per minute (RPM). The emulsion was then allowed to cool to room temperature of 25°C with gradually stirring. The fragrance was then added to the emulsion and then stirred until homogeneity was achieved. It was then covered and left for 12 hrs for the moisturizing cream to set, after which it was more vigorously stirred again at a high speed of 2500 revolutions per minute (RPM) to improve homogenization. The emulsion, which is the moisturizing cream, was then poured into a suitable airtight plastic container for storage and analysis. The same procedure was followed for producing with mineral oil.

2.2.1 Mineral composition of the D. regia oil and mineral oil cream: Delonix regia seed oil cream and mineral oil cream were analysed for their mineral content using Atomic Absorption Spectrophotometer, (Model 215 VGP BUCK Scientific). The samples analyses were based on standard methods of AOAC (2016). Metals such as magnesium, lead, iron, zinc, potassium, and sodium were determined. Accurately weighed (2.0g) of the cream samples were transferred into a silica crucible and kept in a muffle furnace for ashing at 550°C for 3 hours and then 5ml of 6M HCl was added to the crucible. Then, the crucible containing the acid solution was kept on a hot plate and digested to obtain a clear solution. The final residue was dissolved in 0.1M HNO₃ solution and made up to 50mL with distilled water. The digested samples were transferred to the Atomic Absorption Spectrophotometer (AAS). Working standard solutions were prepared by diluting the stock solution.

2.2.2 Physical and Chemical Properties of Moisturizing Cream Produced from Delonixregia Seed Oil and Mineral oil at Different Storage Period: The physical and chemical properties such as pH, moisture content, non-volatile matter, specific gravity and total fatty matter of the D. regia oil were determined according to the methods of Ekpunobi et al., (2014) with some modifications. The physical and chemical properties of the moisturizing cream were studied immediately after production, a year after production, two years after production and three years after production noting that the shelf life of amooisturizing cream is usually three years.

3. RESULT AND DISCUSSION

3.1 Physicochemical properties of the oils

The physical and chemical properties of the D. regia oil are shown in Table 1. The specific gravity obtained for the D. Regia seed oil was 0.99 and 0.87 for mineral oil. This value was very close to those obtained for non-conventional seed oils reported by Olatidoye et al., (2010) and Atasie et al., (2009) and 0.8548 for paraffin oil reported by Oyedeji et al., (2010). The pH (> 10)

value. D. regia is slightly higher than the value (6.88) reported by Alemu et al., (2015) while the pH of mineral oil is slightly lower than that of paraffin oil (7.97) reported by Oyedele et al., (2010). This is likely due to the variation in the species, environmental and soil conditions. Similarly, acid value (22.44 mg KOH/g) is higher than 10.40 mg KOH/g and 20.50 mg KOH/g reported by Adewale (2010) and Oyedele et al., (2017), respectively. The peroxide values for the D. regia seed oil was 7.0 mEq/kg. This is an indication that the oil was freshly extracted and prepared. Low peroxide value indicates high resistance to peroxidation and low rate of spoilage, Nielsen (2003). Rancidity is noticeable when the peroxide value is well above 10 mEq/kg, Olatidoye, (2010). The iodine value obtained suggests the presence of saturated fatty acid and could be used to quantify the number of double bonds present in the oil which also reflects the susceptibility of oil to oxidation. Dregia seed oil has low saponification value. It has been reported by Mestrallet et al., (2004) that oils with high saponification values contain a high proportion of short chain fatty acids, and thus D. regia seed oil contains no short chain fatty acids.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Delonixregia seed oil (DRSO)</th>
<th>Mineral Oil</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific gravity (g/cm³)</td>
<td>0.99</td>
<td>0.87</td>
</tr>
<tr>
<td>pH</td>
<td>10.70</td>
<td>7.01</td>
</tr>
<tr>
<td>Colour</td>
<td>Light green</td>
<td>Colorless</td>
</tr>
<tr>
<td>Viscosity</td>
<td>47.60</td>
<td>11.52</td>
</tr>
<tr>
<td>Solubility in H₂O</td>
<td>Partially miscible</td>
<td>Partially miscible</td>
</tr>
<tr>
<td>Refractive index</td>
<td>1.4335</td>
<td>1.5246</td>
</tr>
<tr>
<td>Acid value (mg KOH/g)</td>
<td>22.44</td>
<td>-</td>
</tr>
<tr>
<td>Peroxide value (mEq/kg)</td>
<td>7.0</td>
<td>-</td>
</tr>
<tr>
<td>Iodine value</td>
<td>37.56</td>
<td>-</td>
</tr>
<tr>
<td>Saponification value (mg KOH/g)</td>
<td>30</td>
<td>-</td>
</tr>
<tr>
<td>Unsaponifiable matter</td>
<td>2.81</td>
<td>1.02</td>
</tr>
</tbody>
</table>

3.2 Mineral constituents of the moisturising creams

Selected minerals investigated in this study for D. regia seed oil and mineral oil cream were magnesium, lead, iron, zinc, potassium, and sodium (Table 2). The cream samples do not contain cadmium but contain a trace of lead. Many studies have shown that low level of lead is permitted in cosmetics. High level of lead in cosmetics preparation can be harmful as it can be absorbed through the skin and studies on the absorption of lead by humans damage the human skin Sun et al., (2002) & Filon et al., (2006). pH and metallic chemical structure are likely factors that could enhance percutaneous penetrations of metals Filon et al., (2006) and Hostynék et al., (2003).

Tsankov et al., (1982) determined lead contents in various cosmetics products and personal body care products. The majority of cosmetics products contained lead approximately 2.08 ppm. Based on the sub-acute dermal toxicity study on albino rats, Tsankov et al., (1982) proposed that the maximum allowable concentration of lead should not be more than 10 ppm.

<table>
<thead>
<tr>
<th>Elements</th>
<th>D.regia Seed Oil Cream(ppm)</th>
<th>Mineral Oil Cream</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg</td>
<td>7.5632</td>
<td>7.6791</td>
</tr>
<tr>
<td>Pb</td>
<td>1.028</td>
<td>1.8210</td>
</tr>
<tr>
<td>Fe</td>
<td>BDL</td>
<td>0.05</td>
</tr>
<tr>
<td>Zn</td>
<td>1.5643</td>
<td>1.8785</td>
</tr>
<tr>
<td>K</td>
<td>0.0857</td>
<td>2.4650</td>
</tr>
<tr>
<td>Na</td>
<td>7.1432</td>
<td>9.4560</td>
</tr>
</tbody>
</table>

BDL: Below Detection Level

3.3 Physicochemical properties of the moisturising creams at different storage period

The physical and chemical properties of Delonix regia seed oil cream and mineral oil creams at different storage period are as shown in Figures 1-4. The results showed that the pH of D. regia seed oil increased as the storage period increased while the pH of mineral oil cream decreased as the storage period increased, this indicates the amount of free fatty acids, and bases present in each of the creams, namely, D.regia seed oil cream and Mineral oil cream. An increase in pH value may occur if alkaline metal oxides bind acids and thereby form metallic soap which may also cause further saponification. This process, however, will not influence the shelf life of the moisturizing cream or the quality (Lautenschlayer, 2016). Ideally, the pH of an emulsion should be close to the pH of the human skin which is 5-7 Oyedjie et al., (2011). The total fatty matter for D. regia seed oil decreased as the storage period increased while that of mineral oil cream increased as storage period increased. Total fatty matter maintains and nourishes the dry skin. Hence, it is one of the most important factors and ingredient that determines the quality of moisturizers, Pratibha et al., (2016).

The non-volatile matter for D. regia seed oil increased as storage period increased while that of mineral oil cream decreased as storage period increased. The non-volatile matter is the substance remaining after removal of water by the process of heating and evaporation, Pratibha et al., (2016). These emulsions have high non-volatile matter which indicates that they contain ample amount of waxes and other substances with high boiling points.

The specific gravity for D. regia seed oil decreased as the storage period increased while for mineral oil, it increased as storage period increased. This, therefore, indicates that specific gravity influences the choice of the material especially oils to be used in formulating moisturizing creams.
The moisture content for *D. regia* seed oil increased as storage period increased while for mineral oil, it decreased as storage period increased. Most cosmetic samples have high percentage moisture content (50-90) and thus will provide adequate moisture and hydration to the skin thereby making the skin feel soft, Oyedeji and Oderinde (2005).

Finally, the cream was stable in terms of texture, color and odor within the three (3) - year period of storage.

![Fig. 1: pH of the moisturising creams produced at different storage period](image1)

**DRSOC - Delonixregia Seed Oil Cream**  
**MOC - Mineral Oil Cream**  

![Fig. 2: Non-volatile matter of the moisturising creams produced at different storage period](image2)

![Fig. 3: Specific gravity of the moisturising creams produced at different storage period](image3)
4. CONCLUSION
It is evident in this study that duration of storage has an effect on the physicochemical properties of moisturising creams with respect to the type of oil, namely, fatty oil or petroleum mineral used in production. Despite the fact that the physicochemical properties varied with an increase in storage period, the creams made from the two sources of oil still fell within the acceptable standard values as prescribed by most regulatory bodies in the world such as Standards Organization of Nigeria (SON). Therefore, the two moisturising creams still retained their quality during the three (3) year period of storage.

5. REFERENCES


