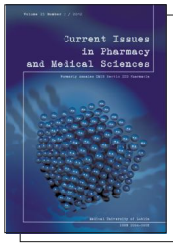

2012 © Curr. Issues Pharm. Med. Sci. Vol. 25, No. 2

Current Issues in Pharmacy and Medical Sciences

Formerly ANNALES UNIVERSITATIS MARIAE CURIE-SKLODOWSKA, SECTIO DDD, PHARMACIA
on-line: www.umlub.pl/pharmacy

Physical and chemical properties of cosmetic cream made of ingredients obtained from *Juglans regia* L.

KATARZYNA WOJCIECHOWSKA*, MARIA ZUŃ, DOROTA DWORNICKA, ANNA SEREFKO, KATARZYNA ŚWIĄDER, EWA POLESZAK

Chair and Department of Applied Pharmacy, Medical University of Lublin, Poland

ABSTRACT

The aim of the study was obtaining the cosmetic cream containing walnut oil, defatted walnut seeds and gelling agents like beeswax and witepsol. The walnut oil and defatted walnut seeds were received by extraction method using chloroform: methanol (1: 1), (v/v). Then the physical and chemical properties of the cream were investigated. The consistency, drop point and flow temperature, the spreadability and rheological properties in the temperature range 22°C -50°C were estimated. The release of the model substance, 4-aminobenzoic acid, in the same range of temperatures, was defined as well. Previously, no studies had been conducted, in which the physicochemical properties of semisolid preparations with walnut oil and defatted walnut seeds would be estimated.

Keywords: cosmetic cream, walnut oil, 4-aminobenzoic acid, Juglans regia

INTRODUCTION

There are about 15 types of Juglans, which belong to Juglandaceae group. Juglans regia originates from the Near East and now it is widely cultivated in Europe, North Africa and North America, too.

The walnut seeds are a high-energy product, which contains about 650 kcal per 100g. The reason for this is a large amount of fats in seeds approx. 63.6-67.2%. The seeds oil contains 10% saturated fatty acids (palmitic acid and stearic acid) and 90% unsaturated fatty acids, out of which 9-30% is oleic acid, 57-76% is linolic acid and 2-16% is linolenic acid [22]. The walnut oil is a rich source of tocoferol, which occurs in four forms. Beta and gamma tocopherols consist of 19.5mg/100g followed by delta tocopheroles – 3.05mg/100g and alfa tocopheroles 2.6mg/100g [12].

Lipophilic compounds like lipids, triacyloglicerides, tocopheroles are separated by the use of nonpolar solvents like metanol, aceton, chloroform or their mixtures [4, 6]. Walnut oil may be obtained by using cold press machines [28].

Nowadays, there is a great interest in making semisolid formulations for dermatological use due to their nourishing, moisturizing, and protective properties [15, 17, 20]. In current literature, the most popular products used in cosmetics are avocado oil [5], almond oil [2] and other like macadamia nut oil [3], jojoba wax [25], shea butter [21].

There is some information about possibilities of using lipophilic and hydrophilic fractions obtained from Juglans regia, which can be used in cosmetic formulations [26, 27] but there is not enough information regarding the use of defatted matter obtained from Juglans regia as a gel agent. Apparently, the use of the walnut oil for cosmetic purpose is due to its high content in essential fatty acids, especially linoleic and linolenic acids. These acids are considered crucial agents of the most important function of the skin responsible for the regulation of the transepidermal water loss [26].

The main investigation to determine the properties of semi-solid preparation is rheological measurements (plastic features, spread capacity and tixotropic properties) [9, 13, 18]. The rheological properties of an emulsified system significantly determine its usefulness and purpose, so they are needed in the analysis of its properties [17]. Other important investigations of semi-solid preparations for dermatological use are extensometry properties [1, 11, 16], determination of the drop point and the flow temperature and the release of a model drug from them [8, 19, 10].

Based on the collected information and data, a cream has been prepared. This cream contains, as the main ingredient, walnut oil and defatted powder of walnut seeds. Then, physical and chemical properties of the cream have been determined.

MATERIALS AND METHODS

Raw material treatment and method of extraction

The seeds of Juglans regia were collected in the South of Poland in the autumn of 2008.

The walnuts were stored until the day of experiment at 10°C. On the day of experiment, the walnuts were stripped of husk, crushed, and sieved. After that, an amount of 1000g of walnut was placed in percolator, wetted with mixture of chloroform and methanol solvent 1:1 (v/v). The walnuts re-

Corresponding author
* Chair and Department of Applied Pharmacy,
Medical University of Lublin, 1 Chodzki Str., 20-097 Lublin, Poland
e-mail address: kgierucka@wp.pl

mained in percolator for 24h and on the next day, percolation process was performed. The process was repeated three times. Next, the solvents were distilled from the liquid extract in the rotary evaporator under lowered pressure and then the oil was separated from the mixture.

The extracted and defatted seeds were dried and powdered. The powder was semi-yellow, without smell. It was not soluble in water and other solvents but expanded when mixed with water.

Formula and cream preparation

Defatted, powdered walnut seeds	1.5
Water	11.0
Beeswax	4.0
Walnut oil	48.0
Witepsol	21.5

From the defatted, powdered walnut seeds, water and walnut oil, an emulsion was prepared, which was modified with witepsol and white beeswax in order to improve its gel structure. To the final phase 5% of weigh of 4-aminobenzoic acid was introduced as a model drug.

RESULTS AND DISCUSSION

Physical and chemical investigation of obtained cream

a) Determination of consistency and color

The formulation had a cream consistency, brown color and a smell of walnut.

b) Determination of flow temperature and drop point

Drop point and flow temperature of the cream determined by Ubbelhod's apparatus [14] were 30°C and 32°C.

c) Determination of extensometry

The spreadability of the cream was measured with an extensometer [11]. The apparatus consists of glass plate fixed in a holder and a second glass plate. The sample of 1 cm^3 of cream was applied into the centre of the larger glass plate and the second plate was put on the sample. The weight of 1200g was placed on the upper glass plate. After 8 minutes, the diameter of the spread sample was measured. The experiments were performed in triplicate and mean diameter was calculated. The spreadability for the cream was 24.44cm^2.

d) Rheological properties of the base

Rheological properties of the cream formulations were conducted at Rheotest 2- Medinger apparatus using cylinders s/s1, with sheer rate (Dr) in the range (1.5s^{-1}- 656s^{-1}). The results of the experiment are shown in Fig. 1, 2, 3a and 3b.

The obtained values of shear stress (Tr) obtained at increasing values of shear rate (Dr) at temperature 22°C enable us to draw ascending curve characteristic for plastic flow. Measurements of shear stress Tr were determined at decreasing values of shear rate. The values of shear stress (Tr) enable us to draw the second descending curve, which has a common start and end point with the ascending curve.

Resultantly, the close hysteresis loop area was obtained having the field magnitude of 236657x10-1Pa, (fig. 1).

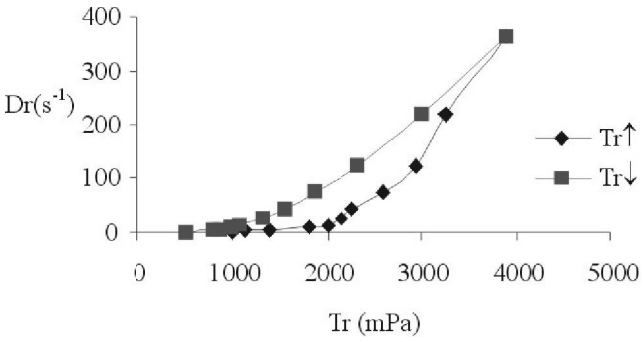

Figure 1. Ascending and descending rheogram for cream at temperature 22°C

Taking the same measurements for cream at the temperature 30°C, curve characteristic for pseudoplastic flow was obtained (fig. 2). At the same temperature, the product had thixotropic properties. Hysteresis loop area had the field magnitude of 207586x10-1Pa, which is smaller than the previous one. It indicates that when the temperature increases, the thixotropic properties decrease. At the temperature above 30°C, the product did not show thixotropic properties.

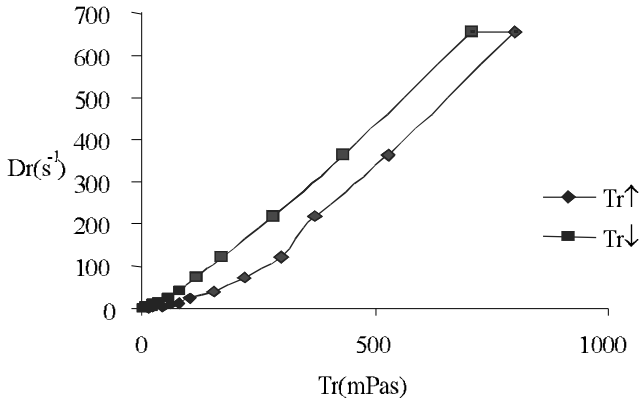

Figure 2. Ascending and descending rheogram for cream at temperature 30°C

In addition, the effect of viscosity change at the temperature range of 30°C-50°C was evaluated. The results are showed in Fig. 3a and 3b.

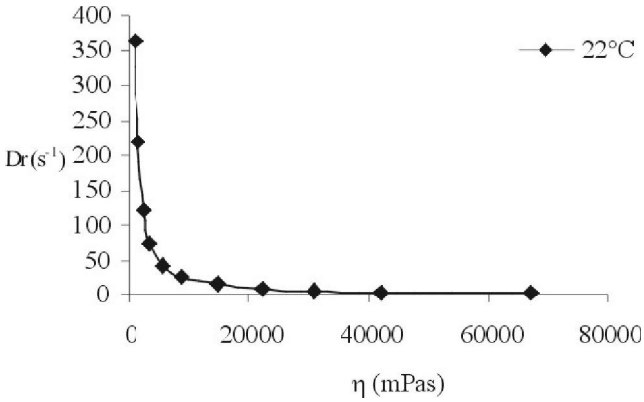

Figure 3a. Effect of viscosity change in relation to shear rate at temperature 22°C

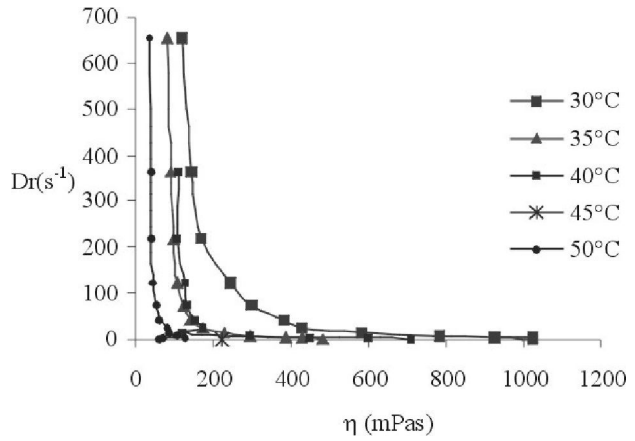

Figure 3b. Effect of viscosity change in relation to shear rate at temperature 30°C, 35°C, 40°C, 45°C, 50°C

The presented figures show that when the temperature increases, the character of flow of the cream will change from plastic flow into pseudoplastic flow and to newton flow.

Finally, the release of 4-aminobenzoic acid (PABA) was determined. PABA was used as a model drug. It adsorbs radiation with the wave length of 280 -320nm (range of UVB) and it can be used as antisolar formulation [23, 24].

A release test of PABA was performed with Dissolution Paddle Apparatus and the extracting cell was described in Farmakopea Polska VIII [10, 14].

The release of PABA to water was conducted at the temperatures 22°C, 30°C, 35°C, 40°C, 45°C, 50°C. The amount of released substances after predetermined time was determined with spectroscopic method. Using modified Higuchi equation, the constant rate of release (K) and lag time (Td) were calculated.

The amount of released substances of cream formulation at constant rate of release (K) and lag time (Td) are presented in Table 1.

Table 1. The amount of released substances from cream formulation (Mt), constant rate of release (K), and lag time (Td)

Temp. (°C)	t(h)	Mt (mg)	K* ($mgh^{1/2}$)	Td (h)
22	9	6.01	2.20	2.85
30	9	13.62	5.72	0.70
35	9	17.67	6.37	0.04
40	9	30.56	12.57	0.64
45	9	45.99	18.97	0.56
50	9	77.18	30.70	0.30

CONCLUSION

Defatted walnut seeds and oil fraction of walnut was used in preparation of cream formulation. Addition of white beeswax and witepsol reinforced cream to get a gel structure. The cream had creamy consistency and the application on the skin was very easy. The cream was similar to vanishing cream; it means that when applied on the skin, the contact surface remained shiny and after certain time (2-3 hours), it had a tendency to reduce its fattiness. The usefulness of this cream is determined by the following test results: drop point 30°C, flow temperature 32°C, spreadability 24.44cm^2 under the load of 1200g. The cream at room temperature has a plastic flow and thixotropic properties. Thixotropic properties were observed up to the temperature of 30°C. With the increase of temperature, the plastic flowing of the cream changed into pseudoplastic flow and then newtonian flow. The release of 4-aminobenzoic acid from the cream increased with the increasing temperature from 2.3% at the temperature 22°C to 32% at the temperature 50°C. The substance was later released from the cream at 22°C; at other temperatures, the lag times were very close.

REFERENCES

1. Achim M., et al.: Topical dosage forms containing fluidextract of Ruscus aculeatus: Formulation, preparation and physical characterization. *Clujul. Medical.*, 83, 99, 2010.
2. Akhtar N. et al.: Formulation and in vitro evaluation of a cosmetic emulsion from almond oil. *Pak. J. Pharm. Sci.*, 21, 430, 2008.
3. Akhtar N., Yazan Y.: Formulation and in-vivo evaluation of cosmetic multiple emulsion containing vitamin C and wheat protein. *Pak. J. Pharm. Sci.,* 21, 45, 2008.
4. Amaral J.S. et al.: Triacylglycerol composition of walnut (Juglans regia L.) Cultivars: Characterization by HPLC -ELSD and Chemometrics. *J. Agric. Food Chem.*, 52, 7964, 2004.
5. Ballamann C., Mueller BW.: Stabilizing effect of cetostearyl alcohol and glyceryl monostearate as co-emulsifiers on hydrocarbon-free O/W glyceride creams. *Pharm. Dev. Technol.*, 13, 433, 2008.
6. Bao-Yuan Guo et al.: Separation and determination of phpspholipids in plant seeds by nonhydrous capillary electrophoresis. *J. Chrom.*, 1074, 205, 2005.
7. Behagel O., Rothman S., Schultze W.: Beziehugen zwischen selektiver Ultrafiolett absorption und chemischer Konstitution. *Strahlentherapie*, 28, 110, 1928.
8. Bonferoni M.C., Rossi S, Ferrari F., Caramella C.: A modifies Franz diffusion cell for simultaneous assessment drug release and washability of mucoadhesive gels. *Pharm. Dev. Technol.*, 4, 45, 1999.
9. Bucur L., Hirjau V., Istudor V.: *Elaeagnus angustifolia L. flowers soft extract valorification in a dermatological preparation rheological control.* Farmacia, 2009.
10. Bottan F., Di-Colo G., Namüpieri E. et al.: Influence of drug concentration on in vitro release of salicylic acid from ointment bases. *J. Pharm. Sci.*, 63, 1979, 1974.
11. Ceulemans J., Santvliet Van L., Ludwig A.: Evaluation of continuous shear and creep rheometry in the physical characterization of ointments. *Int. J. Pharm.*, 176, 187, 1999.
12. Delgado M.M., Bustamente-Rangel., Sanchez-Perez A., Pressurized liquid extraction prior to liquid chromatograpy with electrochemical detection for analysis of vitamin E isomers in seeds and nuts. *J. Chrom.*, 1056, 249, 2004.
13. Eccleston G.M.: Structure and rheology of cetomacrogol creams: the influence of alcohol chain and homologue composition. *J. Pharm. Pharmacol.*, 29, 157, 1977.
14. Polish Pharmacopoeia VIII, Vol. I-III, 2008.
15. Gloor M., Bettinger J., Gehring W.: Modification of stratum corneum quality by glycerin-containing external ointments. *Hautarzt.*, 49, 6, 1998.
16. Hadi I.A., Ugrane H.E., Farouk A.M., Shayoub M.: Formulation of polyethylene glycol ointment bases suitable for tropical and subtropical climates. Acta Pharm. Hung. 59, 137, 1989.
17. Jimenez-Soriano M.M., Fresno- Contreras M.J., Selles F.: Development of a cream from a self-emulsifying base and moisturizing actives. *II Farmaco*, 56, 513, 2001.
18. Korhonen M., Niskanen H., Kiesvaara J., Yliruusi J.: Determination of optimal combination of surfactants in creams using rheology measurements. *Int. J. Pharm.*, 197, 143, 2000.

19. Lalor C.B., Flyn G.L., Weiner N.: Formulation factors affecting release of drug from topical formulation. 1. Effect of emulsion type upon in vitro delivery of ethyl p-aminobenzoate. *J. Pharm. Sci.* 83, 1525, 1994.
20. Moldavian M., Ciortea L.: Efficacy evaluation of different cream formulations on healthy skin properties. *Farmacia*, 58, 787, 2010.
21. Onwuchekwa V.: Shea butter in cosmetics. *Drug Cosmet. Ind.,* 131, 42, 1982.
22. Ozcan Mahmed Musa: Some nutrional characteristics of friut and oil of walnut (*Juglans regia* L.) Growing in Turkey. *Iran J. Chem. Eng*., 28, 57, 2009.
23. Rothman S., Rubin J.: Sunburn and paraaminobenzoic acid. *J. Invest. Dermatol*. 5, 445, 1945.
24. Salvador A., Chisvert A. Rodrigez A., March J.G: Indirect spectrophotometric determination of p-aminobenzoic acid in sunscreen formulation by sequential injection analysis. *Anal. Chem. Acta*, 493, 233, 2003.
25. Taguchi M., Kunimoto T.: Jojoba wax in cosmetic creams and lotions. *C&T*, 95, 39, 1980.
26. Tsamouris G., at al.: Lipid analysis of Greek walnut oil (*Juglans regia* L.) *Z. Naturforsch*., 57c, 51, 2002.
27. Walnut seed meal extract US Patent 6395261
28. Zwarts L., Savage G.P., Mc Neil D.: Fatty acid content of New Zeland – grown walnuts (*Juglans regia* L.). *Am. J. Chin. Med*., 25, 175, 1997.