

The Influence of Packaging on Cosmetic Emulsion during Storage Assessed by FT-NIR Spectroscopy and Color Measurements

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Synopsis

FT-NIR spectroscopy and color measurements were applied to evaluate the influence of packaging on cosmetic cream stored at room and refrigerated conditions. Commercial cosmetic cream was stored for 2 and 4 mo in five containers intended for cosmetics: aluminum jar with polypropylene thermos (Al/PP), acrylic jar with polypropylene thermos (Ac/PP), glass (G) jar, polypropylene (PP) jar, and styrene acrylonitrile (SAN) jars. Principal component analysis (PCA) of the FT-NIR spectra showed the effect of time on tested samples stored in all applied packagings; separate groups were formed by fresh samples and samples stored for 2 and 4 mo. The changes in samples stored in the cold for 2 mo were similar for all applied packagings as compared with fresh samples, although samples stored in SAN jars formed a separate group. After 4 mo, a separate group was formed by samples stored in G jars. For samples stored at room temperature, the influence of packaging material on cosmetic emulsion was clearly visible; four separate groups (Al/PP with PP, Ac/PP, G, and SAN) were created by samples stored for 4 mo. Using partial least squares (PLS1) regression, it was found that the FT-NIR spectra of tested samples correlated with their lightness L^* , a^* parameter, and total color difference ΔE^*_{ab} . FT-NIR spectroscopy is a rapid technique which could be useful to make the best choice of packaging for cosmetics protecting the original quality of products during long-term storage.

INTRODUCTION

Cosmetic products are commodities commonly used by people for skin maintenance, beauty, and treatment of skin diseases in their daily life (1). The use of appropriate packaging system is not only intended for passive protection against humidity, light, and oxygen, but also to improve the overall quality characteristics of their contents, e.g., to extend the shelf-life and enhance the safety or protect sensory properties of products (2–4). Because of the dynamics of the changes on cosmetic market, development of new products should be fast, and accordingly, stability prediction is usually performed by accelerated storage conditions (5). Temperature variation is the main parameter used to induce

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rapid chemical and physical alterations in formulations, which are usually detected by quantification of some components over time (6). Moreover, the kind of cosmetic packaging may also have great importance. The permeability of packaging materials is of great interest because this feature may change essential characteristics of products because of the reaction with environmental oxygen and carbon dioxide (7–9). The development of new materials, mainly polymers, and new fabrication methods have led to a substantial improvement in the quality and versatility of packaging materials (10). The use of appropriate types of packaging materials can improve the stability of cosmetics.

There are few studies on the effect of packaging material on the stability of cosmetics. Available studies concern rather migration of some compounds from packaging to the product, which could be harmful to the consumer health (11), or the influence of cosmetic formulation on some properties of packaging materials (12). Santoro et al. (13) studied the stability of an emulsion containing ultraviolet A (UVA), ultraviolet B (UVB), and infrared sunscreens after storage in different packaging materials (glass and polypropylene flasks; polypropylene and aluminum tubes), but they observed no significant changes in the physical and chemical stability of emulsions stored in different packaging materials.

In recent years, Fourier transform near-infrared (FT-NIR) spectroscopy has found increased application in food, pharmaceutical, cosmetics, petrochemical, chemical, textile, polymer, and other industries (14–16). The NIR spectrum of a product is like its “chemical fingerprint.” It could be applied to obtain qualitative (e.g. origin, type, and adulteration) or quantitative information (e.g. concentration of analyte) about a product. Principal component analysis (PCA) is commonly used for multivariate data reduction with minimum loss of information and to identify patterns in data, showing their similarities and differences. Multivariate calibration methods such as partial least squares (PLS) regression are used to look for the relationship between multivariate data, e.g., FT-NIR spectra, and other parameters of a product. Possible calibration models can be used for the prediction of analyte concentration or parameters of a product from its FT-NIR spectrum (17,18).

Studies concerning the application of FT-NIR spectroscopy for the evaluation of the effect of packaging materials on the overall quality of products are limited. They concern rather food products. Aday and Caner (19) compared the effect of three types of coatings, i.e., chitosan (polysaccharide), whey protein isolate, and shellac (lipid resin), on maintaining the quality of fresh cherry during storage. They used various measurements, including FT-NIR spectroscopy, and found that FT-NIR spectra are useful in predicting the sugar content and firmness of cherries.

Colour measurements are very often used to monitor positive or negative alterations occurring in products, e.g., during storage; however, such measurements were not used to study the effect of packaging materials on cosmetic products.

Therefore, in the present study, FT-NIR spectroscopy and color measurements were proposed as tools to evaluate the influence of various packagings on the overall quality of cosmetic creams stored at refrigerated and room temperatures for 2 and 4 mo. PCA and PLS1 analyses were performed on multivariate data of cosmetic emulsions (FT-NIR spectra) to visualize the changes in creams during storage and to correlate them with the CIE (Comission Internationale de l'Eclairage) 1976 $L^*a^*b^*$ color parameters of creams, respectively. It is highly desirable to use the packaging that protects the original quality of a product during long-term storage. To the best of our knowledge, there are no studies concerning the usage of FT-NIR spectroscopy with chemometric analysis for testing the

overall changes in cosmetic emulsions stored in various packagings and for making the best choice of packaging protecting the original quality of cosmetic products during long-term storage.

MATERIALS AND METHODS

MATERIALS

Moisturizing body creams with shea butter and cocoa, packed in opaque plastic containers, were purchased from a local cosmetic store. They were from the same production batch to be sure that repacked samples will be uniform at the beginning of storage. The cosmetic emulsion was repacked into five different cosmetic containers (30 ml) and stored in a refrigerator (4°–6°C) and at room temperature (22°–25°C) for 2 and 4 mo with natural access of light. Five samples were analyzed for each type of container. The characteristics of the cream containers, including aluminum jar with polypropylene thermos (Al/PP), transparent acrylic jar with polypropylene thermos (Ac/PP), glass (G) jar, polypropylene (PP) white jar, and transparent styrene acrylonitrile (SAN) jar, are presented in Table I.

METHODS

FOURIER TRANSFORM NEAR-INFRARED (FT-NIR) ANALYSIS

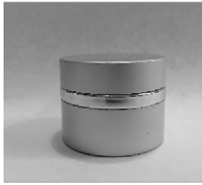




The FT-NIR spectra of the cosmetic cream samples were measured using the MPA (multipurpose analyzer) FT-NIR spectrometer (Bruker Optik, GmbH, Ettlingen, Germany) with an integrating sphere for measuring solids and pastes in diffuse reflection mode. A sample rotator (diameter 45 mm) assuring high reproducibility for heterogeneous samples was applied. The FT-NIR spectra were measured in the range of 12,500–4,000 cm^{-1} . The spectral data of each sample were acquired 32 times with resolution of 16 cm^{-1} and collected using the OPUS software ver. 7.0 (Bruker Optik, GmbH, Ettlingen, Germany).

COLOR MEASUREMENTS

The color of the cosmetic cream samples was determined in the CIE 1976 $L^*a^*b^*$ color space. L^* describes the lightness, a^* indicates the intensity of the red (in the positive range) and the green color (in the negative range), and b^* value indicates the intensity of yellow or blue color in the positive or negative range, respectively. All color functions were calculated for illuminant D_{65} and the 2° angle observer using the tristimulus values obtained using Minolta Chroma Meter CR200 (Konica Minolta, Tokyo, Japan) offering 8-mm-diameter measuring area and wide-area illumination/0° viewing geometry ($d/0^\circ$). The cream samples were placed in granular-materials attachment CR-A50 (Konica Minolta) covered with a quartz plate. For each packaging, five measurements were performed. Total color difference (ΔE^*_{ab}) between the values corresponding to the stored (s) and fresh (f) samples was calculated using the following equation:

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}, \text{ where } \Delta L^* = L_s^* - L_f^*, \Delta a^* = a_s^* - a_f^* \text{ and } \Delta b^* = b_s^* - b_f^*.$$

Table I
Characteristics of Cream Containers

Symbol	Type	Packaging material	
Al/PP	Round jar with thermos	Aluminum jar, polypropylene white thermos and spacer, nut with a protective insert	
Ac/PP	Round jar with thermos	Transparent acrylic jar, polypropylene thermos and spacer, nut with a protective insert	
G	Round jar	Glass jar, nut with a protective insert, without spacer	
PP	Round jar with thermos	Polypropylene white jar, polypropylene white spacer	
SAN	Round jar	Transparent SAN jar, ABS nut with an expanded PE washer	

According to Cserhalmi et al. (20), ΔE_{ab}^* values of 0–0.05 indicate unnoticeable difference, 0.5–1.5 indicate slightly noticeable difference, 1.5–3.0 indicate noticeable difference, 3.0–6.0 indicate well-visible difference, and values higher than 6.0 indicate great difference.

DATA ANALYSIS

PCA with cross-validation of spectral data was carried out on the FT-NIR spectra (12,500–4,000 cm^{-1}) of cosmetic samples. Full cross-validation was also applied to all of the PLS1 regressions. Multiplicative scatter correction (MSC) was used to compensate for multiplicative and/or additive scatter effects in the spectral data. First derivative processing of spectral data was carried out with the Savitzky–Golay procedure, which used a second polynomial order with a 15-point segment (smoothing points). The regression

relationships were evaluated using the coefficient of determination (R^2). The data analysis was carried out using Unscrambler 7.0 (CAMO, Oslo, Norway) software.

Differences between the color parameters of the fresh and stored samples were evaluated by using the least significant difference (LSD) test at $\alpha = 0.05$.

RESULTS AND DISCUSSION

In the present study, the influence of packaging on cosmetic creams stored in different conditions was monitored by FT-NIR spectroscopy and $L^*a^*b^*$ color measurements.

Average FT-NIR spectra of fresh samples and samples stored at refrigerated conditions for 2 and 4 mo and their first derivatives are presented in Figures 1 and 2. No visible changes were observed in the FT-NIR spectra without data processing for samples stored in different packagings both in the cold (Figure 1A) and at room temperature for 2 mo (Figure 2A). Some differences were observed between the first derivative of the FT-NIR spectra of samples stored for 2 mo at refrigerated conditions in SAN jars as compared with other samples (Figure 1A). After 4 mo, a decrease in the intensity of bands at about 7,170 and 5,310 cm^{-1} was observed for all samples as compared with samples of fresh cream (Figures 1B and 2B). It is especially visible for samples stored at room temperature in glass jars (Figure 2B). These bands could be associated with the content of water. A strong water absorption band exists at about 1,450 nm (6,900 cm^{-1} ; OH vibration of water) and at 1,940 nm (5,155 cm^{-1} ; a combination band involving OH stretching and OH deformation) (14). In contrast to other packagings used in the present study, glass jar had packaging without thermos or an additional spacer between the nut and the content of the jar. This could be the reason for the most visible changes in the content of water in the samples stored in this packaging.

PCA was performed on FT-NIR spectra to visualize the changes in cream samples during storage. The PCA results of the FT-NIR spectra of cosmetic emulsions stored in the cold and at room temperature for 2 and 4 mo are presented in Figure 3. The effect of time on cream samples stored in Al/PP, Ac/PP, G, PP, and SAN jars was observed; separate groups were formed by fresh samples (F) and samples stored for 2 and 4 mo.

The first two principal components (PC1 and PC2) described 98% of the total data variance of samples stored both in the refrigerated conditions and at room temperature (Figure 3). The first principal component PC1 that explained 94% (samples stored in the cold) and 92% (samples stored at room temperature) of the total data variance was linked to the time of storage. It differentiated samples stored for 4 mo from fresh samples (F) and those stored for 2 mo. The PC2 explained 4% and 6% of the total data variance of samples stored in the cold and at room temperature, respectively. It was related to the variability due to the type of samples (fresh and stored samples).

It was found that the changes in samples stored in the cold for 2 mo were similar for all applied packaging materials as compared with fresh samples (F), although samples in SAN jars formed a separate group (Figure 3A). After 4 mo, a separate group was formed by samples stored in glass (G) jars. For samples stored at room temperature, the influence of packaging materials on cosmetic emulsions was observed just after 2 mo of storage (Figure 3B). The smallest changes in the FT-NIR spectrum of a cream as compared with the fresh one (F) were observed for samples stored in Al/PP. The changes for other packaging

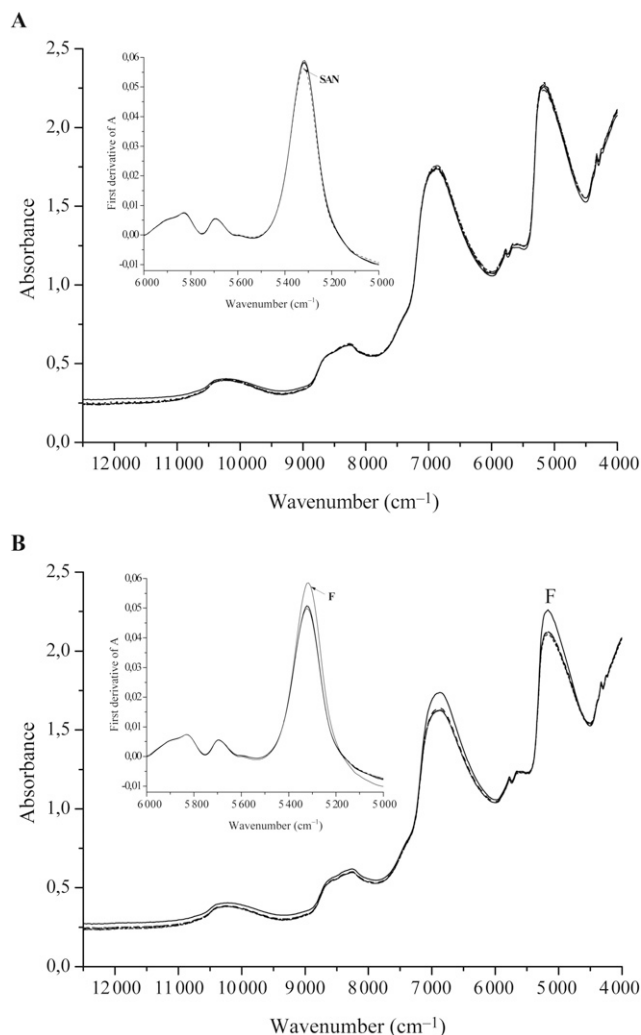


Figure 1. FT-NIR spectra and their first derivatives (inside) of fresh cream (F) and other samples stored in refrigerated conditions for (A) 2 mo and (B) 4 mo.

materials were similar. Four-month storage resulted in the formation of four groups: Al/PP plus PP, SAN, Ac, and G. As it was observed after direct analysis of the FT-NIR spectra, the differences between fresh cream and samples stored at room temperature in glass jars were the most clear. It cannot be excluded that the most visible effect of glass on overall changes in cream samples as compared with fresh samples is due to different structure and properties of glass from those of polymer materials used as packagings in the present study; they formed one group. Moreover, glass jars were packaging without additional thermos or a protecting spacer.

Storage of cosmetics in different packagings may influence the color of products. Therefore, in the present study, color parameters of stored samples were also measured. The results of $L^*a^*b^*$ color measurements and total color differences (ΔE^*_{ab}) of stored samples as compared with the fresh ones are presented in Tables II and III. A slight increase in

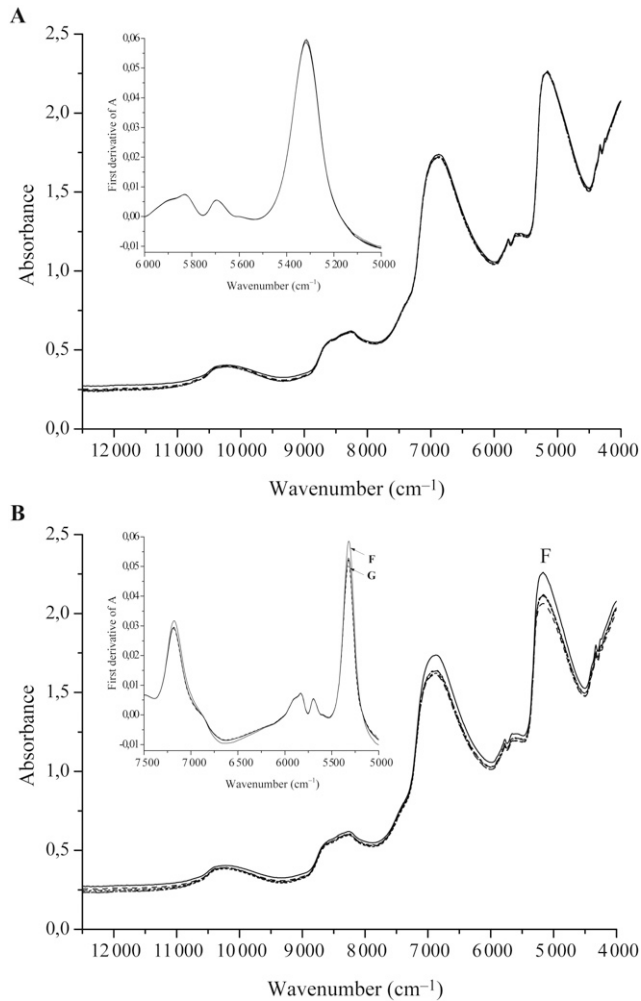


Figure 2. FT-NIR spectra and their first derivatives (inside) of fresh cream (F) and other samples stored at room temperature for (A) 2 mo and (B) 4 mo.

L^* values of samples stored in the cold as compared with that of fresh cosmetic cream was observed, whereas lightness of samples stored at room temperature decreased. The value of a^* and b^* was not significantly changed in the case of samples stored in refrigerated conditions. For samples stored at room temperature for 4 mo, a^* value slightly shifted to redness. It was especially visible for G samples (a^* changed from -2.73 to -1.98). A slight increase in b^* values was also observed for all samples in all packagings (cream became more yellow) with an exception of SAN samples.

Taking into account the values of ΔE_{ab}^* , no visual changes in color of all samples stored in the cold for 2 mo were detected (ΔE_{ab}^* from 0.05 to 0.16). After 4 mo of storage, the color difference between fresh and stored samples could be defined as unnoticeable (for Al/PP Ac/PP, and G) or slightly noticeable (PP and SAN). For samples stored at room temperature, the color difference between fresh and stored sample was slightly noticeable (ΔE_{ab}^* from 0.86 to 1.19 for 2-mo storage, and from 1.02 to 1.46 for 4-mo storage). The

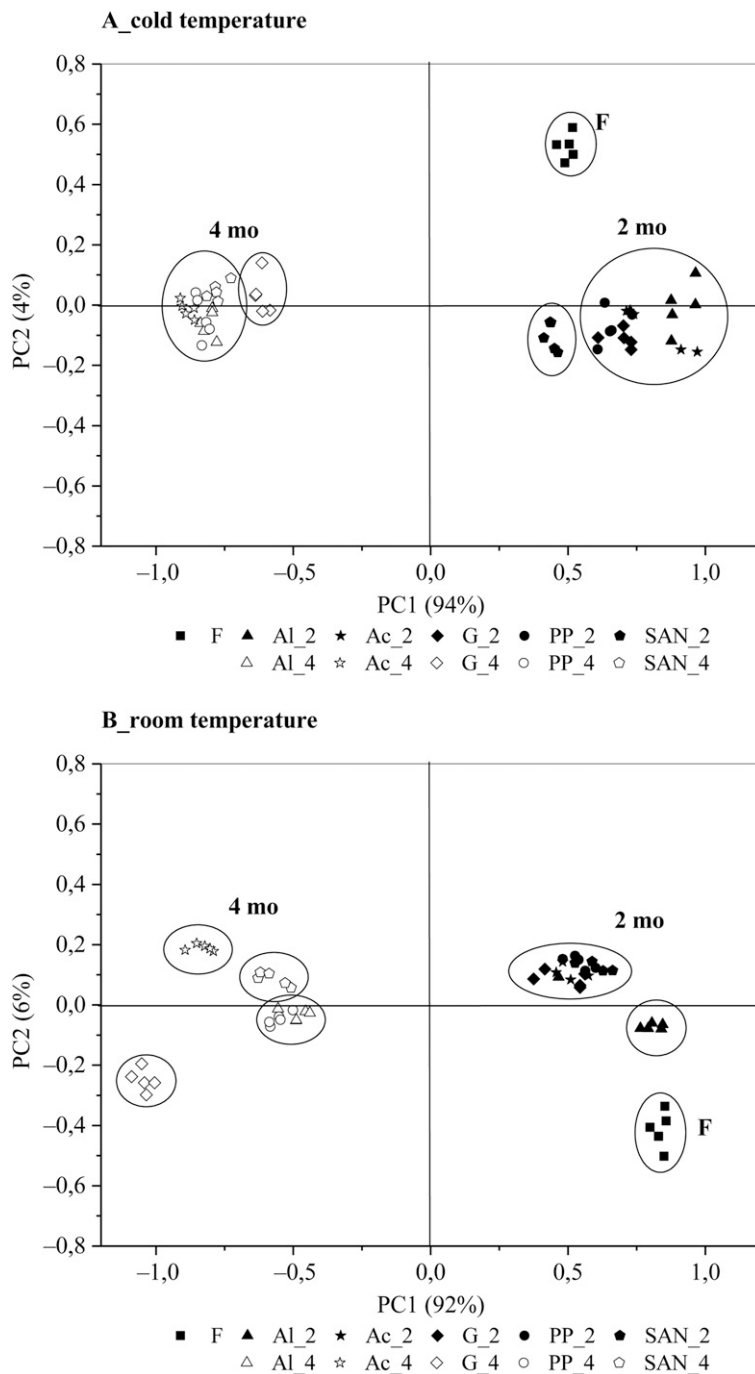


Figure 3. PCA of the FT-NIR spectra of cream samples stored for 2 and 4 mo in different packagings (A) in the cold and (B) at room temperature: F, fresh samples; Al/PP_2 and Al/PP_4, samples stored in aluminium jars with polypropylene thermos for 2 and 4 mo, respectively; Ac/PP_2 and Ac/PP_4, samples stored in acrylic jars with polypropylene thermos for 2 and 4 mo, respectively; G_2 and G_4, samples stored in glass jars for 2 and 4 mo, respectively; PP_2 and PP_4, samples stored in propylene jars for 2 and 4 mo, respectively; and SAN_2 and SAN_4, samples stored in SAN jars for 2 and 4 mo, respectively.

Table II
The L^* , a^* , b^* , and ΔE^* Values of Cream Stored at Refrigerated Temperature

	Month	L^*	a^*	b^*	ΔE^*
F	0	80.50 ± 0.11	-2.73 ± 0.01	2.16 ± 0.04	0
Al/PP	2	80.48 ± 0.12	-2.74 ± 0.01	2.10 ± 0.16	0.16 ± 0.12
	4	80.80 ± 0.02 ^a	-2.83 ± 0.02 ^a	2.18 ± 0.04	0.31 ± 0.02
Ac/PP	2	80.43 ± 0.12	-2.72 ± 0.01	2.18 ± 0.05	0.13 ± 0.04
	4	80.75 ± 0.11 ^a	-2.83 ± 0.04 ^a	2.30 ± 0.02 ^a	0.31 ± 0.09
G	2	80.50 ± 0.03	-2.69 ± 0.01 ^a	2.16 ± 0.01	0.05 ± 0.01
	4	80.82 ± 0.03 ^a	-2.81 ± 0.02 ^a	1.87 ± 0.01 ^a	0.44 ± 0.02
PP	2	80.58 ± 0.08	-2.70 ± 0.03	2.14 ± 0.10	0.15 ± 0.04
	4	81.15 ± 0.05 ^a	-2.87 ± 0.02 ^a	2.27 ± 0.02 ^a	0.68 ± 0.04
SAN	2	80.51 ± 0.07	-2.72 ± 0.03	2.14 ± 0.06	0.09 ± 0.01
	4	80.98 ± 0.03 ^a	-2.88 ± 0.02 ^a	2.15 ± 0.03	0.50 ± 0.03

^aMeans in the column are significantly different from that of fresh sample (LSD test, $\alpha = 0.05$).

highest changes in color were observed for samples stored for 4 mo in G and SAN jars, followed by Ac/PP. It is noteworthy that the changes in FT-NIR spectra were especially visible only for samples stored in G jars.

Taking into consideration all experimental data from FT-NIR spectroscopy and color measurements, it can be concluded that the cream samples changed depending on the packaging type and storage conditions. It is known that the permeation of light and gases through polymer packaging is particularly diverse and sometimes very high (21), therefore not all kinds of polymer packaging can be used for storage of food products and cosmetics. It is also known that polymers, as organic compounds, are characterized by a high coefficient of linear thermal expansion in contrast to many other materials such as glass. Under the influence of temperature, the density of polymers and, as a consequence, other properties associated with it can be changed. Therefore, the effect of temperature must be taken into account when polymer properties are examined. SAN copolymer is a material with high transparency for light, but with good chemical and thermal resistance. Aluminum is the metal used for vacuum metalizing of packaging materials. Originally, this process was used for decorative purposes. Currently, metallization is a widely

Table III
The L^* , a^* , b^* , and ΔE^*_{ab} Values of Cream Stored at Room Temperature

	Month	L^*	a^*	b^*	ΔE^*_{ab}
F	0	80.50 ± 0.11	-2.73 ± 0.01	2.16 ± 0.04	0
Al/PP	2	79.66 ± 0.06 ^a	-2.44 ± 0.02 ^a	2.35 ± 0.13 ^a	0.92 ± 0.03
	4	79.43 ± 0.05 ^a	-2.35 ± 0.03 ^a	2.44 ± 0.08 ^a	1.17 ± 0.03
Ac/PP	2	79.41 ± 0.23 ^a	-2.29 ± 0.01 ^a	2.28 ± 0.10	1.19 ± 0.21
	4	79.34 ± 0.23 ^a	-2.09 ± 0.03 ^a	2.53 ± 0.03 ^a	1.38 ± 0.20
G	2	79.66 ± 0.09 ^a	-2.30 ± 0.01 ^a	2.32 ± 0.05 ^a	0.96 ± 0.07
	4	79.27 ± 0.21 ^a	-1.98 ± 0.02 ^a	2.43 ± 0.02 ^a	1.46 ± 0.04
PP	2	79.72 ± 0.07 ^a	-2.44 ± 0.02 ^a	2.37 ± 0.04 ^a	0.86 ± 0.07
	4	79.70 ± 0.05 ^a	-2.27 ± 0.02 ^a	2.60 ± 0.02 ^a	1.02 ± 0.03
SAN	2	79.56 ± 0.15 ^a	-2.30 ± 0.02 ^a	2.11 ± 0.07	1.03 ± 0.14
	4	79.20 ± 0.08 ^a	-2.12 ± 0.02 ^a	2.19 ± 0.04	1.44 ± 0.07

^aMeans in the column are significantly different from that of fresh sample (LSD test, $\alpha = 0.05$).

Table IV
Coefficients of Determination (R^2) for the PLS1 Regression between FT-NIR Spectra and Colour Parameters of Cosmetic Cream Samples Stored in the Cold and at Room Temperature

Parameter	Mean \pm SD	Method	Factors	R^2_{cal} ^a	R^2_{val} ^a
L^* $n = 105^b$	80.12 ± 0.64	No data processing	4	0.919	0.911
		MSC	5	0.923	0.911
a^* $n = 105$	2.53 ± 0.28	First derivative	5	0.949	0.910
		No data processing	4	0.926	0.918
		MSC	7	0.975	0.931
b^* $n = 105$	2.5 ± 0.18	First derivative	5	0.957	0.924
		No data processing	7	0.724	0.498
		MSC	6	0.684	0.506
ΔE^*_{ab} $n = 105$	0.69 ± 0.49	First derivative	4	0.653	0.461
		No data processing	3	0.851	0.838
		MSC	6	0.890	0.845
		First derivative	3	0.871	0.835

^aCoefficient of determination for calibration (cal) and for validation (val).

^bNumber of measurements.

used method to modify packaging to improve its barrier properties against moisture and gases. As shown in Figure 3B, the samples stored at room temperature in Al/PP were the closest to fresh samples (F). It is probably because of the better barrier properties of this packaging against moisture and gases (at room temperature for at least 4 mo) than other packagings used in the present study. The barrier properties of all packagings during 2-mo storage of cream samples in refrigerated conditions seem to be similar.

Regression analysis using PLS1 was performed to check the possible relationship between FT-NIR spectra ($12,500\text{--}4,000\text{ cm}^{-1}$) and color parameters. The PLS1 regression relationships with no FT-NIR data processing, FT-NIR data after MSC, and the first derivative of FT-NIR data are presented in Table IV. All presented relationships, with an exception of relationship for b^* , had acceptable R^2 for calibration (at least 0.85) and validation (higher than 0.84). The best relationships, expressed as R^2 for validation, were found between L^* or a^* and FT-NIR data used with no spectral data processing (for L^* value) or after MSC modification (for a^* value). The first derivative of FT-NIR data did not improve the R^2 value.

CONCLUSIONS

Altogether, the results of the present study showed that diffuse reflectance FT-NIR spectroscopy and multivariate calculating methods, such as PCA, could be successfully used to determine the effect of packaging materials on overall changes in cosmetic creams during storage. The changes in FT-NIR spectra were probably associated with the changes in the water level in tested samples and they were correlated with their lightness L^* , a^* parameter, and total color difference ΔE^*_{ab} . This study also demonstrated that FT-NIR spectroscopy is a rapid and powerful technique that could be used for quality control of cream emulsions without any sample pretreatment, thus meeting the requirements of “green chemistry.” It could be useful for producers to make the best choice of packaging, protecting the original quality of cosmetic emulsion during storage.

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