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# Characterization and Short-Term clinical study of clay facial mask

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## ABSTRACT

**Clay mineral facial masks are used to treat some dermatological diseases, just for cleansing or reduce the amount of oil secreted by sebaceous glands. There are several types of clays, which vary in mineralogical and chemical composition, color and origin. However, the literature lacks studies involving clay facial masks, in particular regarding their influence on skin´s biomechanical properties. Thus, this work aimed to characterize colored clays and evaluate its influence on skin firmness and elasticity by a short-term clinical study. Different clays (named in this study magnesium aluminum silicate - MAS, white, pink and green) were chemically characterized, and facial mask formulations were prepared. The short-term clinical study was performed through the application of formulations on the skin. The skin firmness and elasticity were assessed before treatment and after mask removal. The statistical analysis showed no significant influence of time or formulations in those parameters, although volunteers reported the sensation of mechanical tension after the removal of the clay facial masks. Thus, the composition of the different clays did not affect the skin viscoelasticity behavior in the short-term clinical study, and a long-term use of this type of formulation must be indicated to observe all the expected benefits.**
Keywords: Clay. Biomechanical properties. Chemical characterization. Facial mask. Clinical study.

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## INTRODUCTION

Clay minerals are used by pharmaceutical or cosmetic industries for several proposes like excipients, due to its rheological properties, or as substances with interesting biological activity in function of its chemical composition (Lopez-Galindo *et al*., 2007; Viseras *et al*., 2007; Zague *et al*., 2007). Among all possibilities, clays are necessary for the cosmetic industry since they present interesting characteristics such as easiness of application and removal, reduced time for drying and hardening, and dermatological innocuousness (Carretero, 2002; Toedt *et al*., 2005).

Their use as a facial mask may be done directly on the skin at room temperature (kaolinite or smectites mixed with water). But, to treat dermatological diseases such as blackheads, spots, acne and seborrhoea it is recommended the application of a mixture comprised of clays and water in a high temperature. The heat increases perspiration and sebaceous secretions while it also opens the pilosebaceous orifices and activates the metabolic change and the excretion of catabolites (Carretero, 2002).

Clay mineral used by the industry typically have a natural origin, although several of these minerals may also be obtained by synthesis. The synthetic clay minerals available in the market has specific properties and uses and has a higher cost compared to natural minerals (Carretero & Pozo, 2009). They belong to the phyllosilicates family of compounds consisting of aluminosilicates containing considerable amounts of K, Mg, Ca, Na and Fe. Also Ti, Mn or Li may be present in smaller quantity. The family of phyllosilicates is divided into various groups of minerals, each group containing several species. In all, there are dozens of phyllosilicates, ranked according to their crystalline structure, chemical (composition, surface chemistry, and charge layer) and physical (morphology and particle size) properties (Bergaya & Lagaly, 2006; Lopez-Galindo *et al*., 2007).

In industrial applications some types of clays can be distinguished: (i) white, (ii) bentonite, (iii) talc, (iv) fibrous clays (palygorskite and sepiolite), and (v) 'common clays' (largely used for traditional ceramic products) which often contain kaolinite and illite/smectite mixed-layer minerals (Bergaya & Lagaly, 2006; Lopez-Galindo *et al*., 2007).

Kaolin (also called 'china clay') is a soft clay that is an essential component in the manufacture of porcelain. Also, it is widely used in the industry of paper, rubber, paint, pharmaceuticals, cosmetics and many other products. Kaolinite is the most common mineral, so kaolin and kaolinite are therefore frequently confused as synonymous terms. Kaolins are usually white, but may also have light shades of brown, cream, yellow, red or gray.

The term bentonite, which is very frequent in the technical vocabulary as well as in different pharmacopeias, is used to designate any plastic, colloidal, swelling clay, basically consisting of a mineral of the smectite group (montmorillonite, saponite or hectorite), with no regard for its origin. Bentonites may have several colors (white, yellow, brown, green, pink, red or black) according to the ions in the smectite structures and according to the associated minerals existing, both related to their origin.

Talc is a clay mineral that chemically is not an aluminosilicate, but a mineral composed of a hydrated magnesium silicate with a crystal structure similar to the smectite structure, but with no layer charge. Talc is usually white or near (greenish or grayish) white. Its particles have a flaky habit and are easily milled, becoming a bright white, unctuous micronized powder. It is widely used in the industries of polymeric and painting products as fillers, and in fine and technical ceramics, pharmaceuticals and cosmetics.

Fibrous clays include two minerals: palygorskite (also known as attapulgite) and sepiolite. Unlike other clay minerals that have a flat crystal habit, sepiolite and palygorskite have a fibrous morphology. These clay minerals occur as fine, white (or near white)-colored particles that are composed of bundles of microscopic fibers. When those particles are micronized, the resulting material is easily dispersed in water and other polar liquids, forming a large volume network of interlaced fibers that traps all the dissolvent, leading to dispersions with high viscosity.

'Common clays' are composed of mixtures of clay minerals (which often contain kaolinite and illite/smectite mixed-layer minerals) with a wide range of associated minerals (quartz, oxides, and hydroxides of iron and aluminum, calcium carbonate, among others). 'Common clays' are largely used only for traditional ceramic products as bricks and tiles (Bergaya & Lagaly, 2006; Lopez-Galindo *et al*., 2007).

Each clay with potential cosmetic applications has a specific dermatological indication that could ideally be related to the type of clay (chemical composition, crystal structure, the presence of associated minerals) and its effect on the skin. These clays are usually identified by their colors by the market. However, information is rare regarding a characterization of each of these colored clay allowing a correlation with dermatological indications. What is commonly found are useful but incomplete information such as yellow clay is used against bacterial infections, red for skin cleansing, blue is effective against acne, green used to reduce the amount of oil secreted by sebaceous glands while black may be indicated for general skin nourishment (Mpuchane *et al*., 2010).

The interest in developing clay facial masks is assigned to cleansing and lifting effects (Khanna & Datta Gupta, 2002). However, the literature lacks studies involving clay facial masks, in particular regarding their influence on skin´s biomechanical properties. This work aimed to characterize colored clays and evaluate the firmness and elasticity of the skin at different time intervals after application of clay facial masks.

## MATERIAL AND METHODS

### Characterization of clay minerals

#### Chemical composition

The qualitative mineralogical composition of commercially available clays (MAS, white, pink and green), purchased from Terramater, was determined using X-ray diffraction (XRD). Clay diffraction patterns were obtained in X-ray diffractometer Bruker AXS D5000. The results were analyzed in software Diffrac Plus version 7.1 and compared with diffraction patterns compiled in the database Powder Diffraction File. A voltage of 40 kV and a current of 40 mA using a CuKα radiation were used. Scans were stepwise from 3° to 65° with a step of 0.05° (2θ) and a step time of 1 s.

Chemical analysis was performed in X-ray fluorescence spectrometer Philips PW2400 XRF. 2.0 g of each clay sample were first dried in porcelain crucibles at 105° C for at least 3 h. Then 10.0 g of 4:1 lithium metaborate: lithium tetraborate commercial flux (Claisse eutectic mixture of 20% lithium tetraborate and 80% lithium metaborate), previously heated to 600° C, was added (Mori *et al*., 1999) to prepare the glass disc used in the analysis. The results were given in percentage for most of the elements and in ppm for those below 1000 ppm.

#### pH values

The pH measurements of the clay dispersions were obtained in pHmeter 8417 Hanna, at 22.0 ± 2.0° C. Each clay was dispersed (1:10) in neutralized distilled water (pH = 7.0) and then filtrated through a qualitative filter paper.

#### Clay facial masks composition

The quantitative composition (% w/w) of the developed base formulation is described in Table 1. All ingredients were pharmaceutical grade, used as received from commercial sources, without any further purification. The magnesium aluminum silicate clay (MAS) was used as gelling, emulsifying and suspending of the formulations. Thus, three different clays (white, pink and green) were incorporated and tested, generating four formulas including

Table 1. Qualitative and quantitative (% w/w) formulation composition. *International Nomenclature of Cosmetics Ingredients (Personal Care Products Council 2016); *Phenoxyethanol; Methylparaben; Ethylparaben; Butylparaben; Propylparaben; Isobutylparaben.

| Ingredients (INCI*) | % (w/w) |
|---|---|
| Magnesium aluminum silicate | 5.0 |
| Xanthan gum | 0.5 |
| Colored clay | 30.0 |
| Glycerin | 4.0 |
| Propylene glycol | 4.0 |
| Preservatives* | 0.5 |
| Distilled water | 56.0 |

the base. The pH of the formulations was adjusted to 5.5 to 6.5 with triethanolamine or citric acid 10% (w/v) according to the need.

**Skin viscoelasticity assessment**

This study was held in open, control intra-individual, complete block and approved by the Ethics Committee (Process CEP 175-06).

Eight healthy Caucasian female volunteers ages 20–30 years (mean ± SD, 25 ± 4.4 years) participated in the study after giving informed consent. Subjects with dermatological abnormalities (e.g., rashes, wounds, scars) in the test areas were excluded. The restrictions imposed on volunteers were: no application of different products in the experimental area, no allergen, anti-inflammatory or vitamin A acid and derivatives treatment during the study. Also, the experimental area should not have contact with clothes during the study or between the start and the end of the measures. Subjects who were pregnant or taking hormone replacement or corticosteroids therapy were also excluded.

The subjects were asked to refrain from using moisturizers on the test sites for 24 h before the study and from applying water to the sites at least 3 h before evaluations. Biophysical measurements were made while volunteers were prone and at least 30 min after acclimation to the room environment (20.0 ± 2.0º C and 40.0 ± 5.0% relative humidity).

An amount of 3.0 g of each formulation were applied randomly on the delimited area of 30 cm2 (5 x 6 cm) on the right or left forearm, on five areas-test and a control area (without formulation) (Bazin and Fanchon 2008). Application time was 20 min, and the removal was performed by paper towel dampened with distilled water.

Skin viscoelastic parameters were evaluated by using a Cutometer® (SEM 575; Courage &Khazaka Electronic GmbH, Cologne, Germany). It measures the vertical deformation of the skin when it is pulled using a controlled vacuum into the circular aperture. The time/strain mode was used with five consecutive cycles of a 5 s suction application followed by a 3 s relaxation period. A 2 mm diameter measuring probe and constant suction of 500 mbar were applied. The skin firmness and elasticity were assessed before treatment (T0) and 20 min, 1 h, and 2 h after mask removal. The parameters M1 (maximum extensibility, meaning skin firmness) and M2 (deformation compared to the initial condition, represents cutaneous elasticity) were expressed at each time of the experiment as an absolute value. The lower M1 and M2 represent the higher cutaneous firmness and elasticity, respectively, and vice-versa (Velasco *et al*., 2014).

**Statistical analysis**

Triplicate data from each site were averaged. The statistical analysis was carried out by longitudinal mixed model, considered as a random effect of the voluntary since formulations and test time were considered fixed.

## RESULTS AND DISCUSSION

**Clay mineral characterization**

The composition of clay minerals influences their properties, and also the pH of the formulation. As shown in Table 2, clays show neutral pH, except for the green one that was around 9.0, due to the presence of calcium carbonate (calcite) in its composition. The mineralogical and chemical compositions of clays are shown in Figure 1 and Table 3, respectively.

Table 2. pH values of clay minerals. Legend: MAS = magnesium aluminum silicate

| Clay | MAS | white | pink | green |
|---|---|---|---|---|
| pH | 6.5 ± 0.5 | 7.0 ± 0.0 | 7.2 ± 0.1 | 8.7 ± 0.1 |

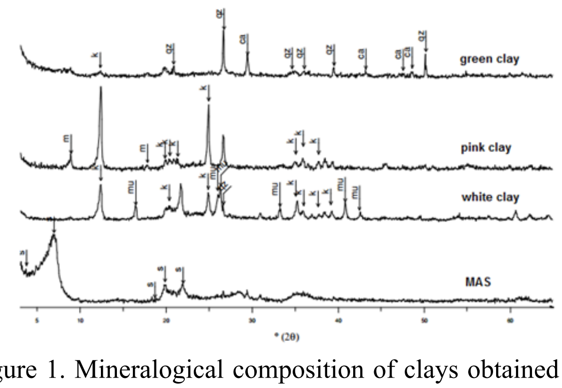


Figure 1. Mineralogical composition of clays obtained by X-ray diffraction. Legend: k = kaolinite; qz = quartz; ca = calcite; m = mica group; mu = mullite; s = smectite group.

Clays are mainly composed of clay minerals but may present associated minerals such as quartz and calcite in function of their natural composition or industrialization processes, becoming residual materials (Besq *et al*., 2003). The XRD pattern of green clay (Figure 1) shows sharp peaks related to quartz and calcite, and also very reduced peaks referring to kaolinite and a mica group mineral. The presence of calcite raises the pH value of its dispersion in water to values around 9.0. Thus, the neutralization of

Table 3. Clay's chemical composition obtained by X-ray fluorescence. Amounts are expressed in % for smaller elements (<0.1) and higher (> 0.1) and in ppm (<1000) for the trace elements. Legend: Loi = loss on ignition; MAS = magnesium aluminum silicate

| Chemical composition | Clay type | | | |
|---|---|---|---|---|
| | MAS | Green | Pink | White |
| $SiO_2$ | 67.05 | 50.80 | 48.63 | 49.76 |
| $Al_2O_3$ | 14.91 | 14.51 | 30.86 | 40.76 |
| MnO | 0.03 | 0.06 | 0.01 | 0.03 |
| MgO | 2.50 | 2.39 | 0.17 | 0.04 |
| CaO | 0.93 | 9.46 | 0.75 | 0.13 |
| $Na_2O$ | 1.59 | 0.02 | <0.02 | <0.02 |
| $K_2O$ | 1.31 | 3.97 | 1.80 | 1.30 |
| $T_iO_2$ | 2.43 | 0.78 | 0.20 | 0.53 |
| $P_2O_5$ | 0.02 | 0.13 | 0.25 | 0.06 |
| $Fe_2O3$ | 1.85 | 5.73 | 6.89 | 1.30 |
| Loi | 6.90 | 12.05 | 9.85 | 4.73 |
| $Na_2O$/CaO | 1.21 | 0.01 | <0.03 | <0.15 |

cosmetic formulations containing green clay is necessary since the skin physiological features slightly acidic pH (5.5 to 6.5) (Rippke *et al*., 2002). After the pH neutralization, calcite is eliminated (releasing carbonic gas) and quartz becomes the main component.

On diffractogram of white clay, characteristic peaks of kaolinite were observed, some associated minerals are also identified: quartz, a mica group mineral (probably illite) and mulite. The identification of mullite is a signal that this clay sample is a mixture of natural clay and a calcined one because mullite is a high-temperature phase obtained by the calcination of white.

The main clay mineral present in pink clay is kaolinite as demonstrated in its XRD pattern. Quartz, a micaceous phase (probably illite) and an iron oxide (hematite) are present as associated minerals in this clay.

The main peaks of MAS's XRD pattern are characteristic of a clay mineral of the smectite group, which confers its peculiar rheological properties. No associated minerals are clearly identified. The MAS clay is designated chemically in the pharmaceutical field as "magnesium aluminum silicate", which corresponds to a mixture of montmorillonite and saponite (both belonging to the smectite group). It was used in this study as gelling, emulsifying, and suspending in the formulations.

Although commercial clays are subjected to purification processes, the green, white and pink clays used in this study presented associated minerals. In the case of the white clay, one of those minerals (mullite) is not natural, indicating probably an industrial contamination of the sample. Unfortunately, the presence of these minerals can negatively impact the clay's properties and also the stability of cosmetic preparations (Besq *et al*., 2003).

Chemical data (Table 3) indicate a high content of CaO (9.46%) on the green clay due to the presence of the mineral calcite in its mineralogical composition. The smectite present in the MAS can be considered Na-Mg type since the chemical analysis showed 2.5% MgO and higher content of $Na_2O$, in relation to the CaO ($Na_2O$/CaO = 1.21). The ratio $Na_2O$/CaO is an important parameter to evaluate the nature of clay minerals of the smectite group, since high ratios of $Na_2O$/CaO (1 to 3) indicate the presence of swelling smectite, while low ratios (less than 1) is typical of non-swelling clay of type 2:1 (Cara *et al*., 2000). Therefore, the ratio equal to 1.21 ($Na_2O$/CaO) justifies the use of MAS as a gelling agent and thickener in cosmetic formulations. The pink clay shows a high content of Fe2O3 (6.89%) due to the presence of iron oxide (probably hematite), responsible for its color. Green clay also has a high iron content (5.73%), but its color cannot be attributed unambiguously to an iron oxide or hydroxide, once the existence of iron green crystal phases is not common and the presence of such phases was not observed in the XRD curve. Green color could also be due to calcite, which can display up green color when contaminated with a serpentine. However, a serpentine also was not identified from XRD pattern. Therefore, likely due to the low crystallinity of the associated phases present in the green clay, which prevents the identification by XRD, it was not possible to explain the reason for its green color unambiguously.

**Clay facial mask efficacy**

Clays are mostly used in facial masks due to their high absorbency levels on the skin surface, such as greases, toxins and even bacteria and viruses (Carretero, 2002). Nevertheless, the tightening effect is also expected for clay facial masks, in function of the product hardening and contraction, after evaporation of water from the preparation, which causes a sensation of mechanical stress (Wilkinson & Moore, 1982; Gaffney, 1992; Carretero, 2002). Also, the chemical and mineralogical composition may influence their effect on the skin. For example, red clay is recommended for dry skin and green clay, for oily skin (Reinbacher, 2002). However, no scientific literature correlates the color of clays with different effects on the skin (Allo & Murray, 2004).

According to the results observed in Figure 2 and 3, the treated and control areas showed slight changes in the M1 and M2 values, at different times. The decrease in M1 and M2 values involves an increase of skin firmness and elasticity, respectively. However, statistical analysis showed no significant influence ($p > 0.05$) of time or test formulations, including the control area, as well as the interaction between these factors, in the elasticity parameter (M2).

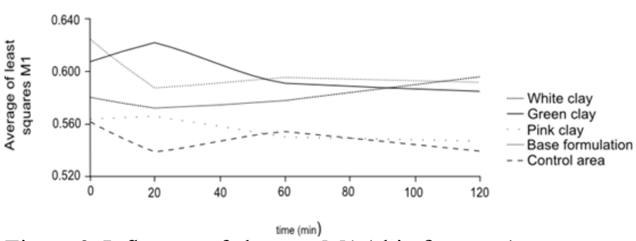


Figure 2. Influence of clays on M1 (skin firmness) parameter.

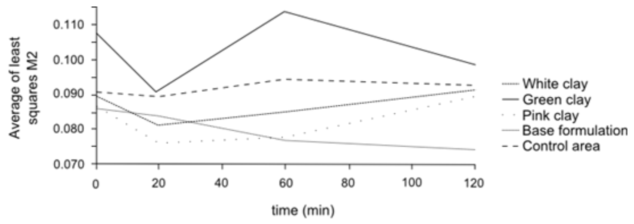


Figure 3. Influence of clays on M2 (cutaneous elasticity) parameter.

Although volunteers reported the sensation of mechanical tension after the removal of the clay facial masks (t = 20 min), the statistical analysis of M1 values showed no significant difference ($p > 0.05$) in comparison with T0. Thus, the nowadays available equipment is not enough to detect the tensor effect felt by volunteers.

The main effect of clay facial masks, in a short-term application, comes from hardening and contraction of the product after the evaporation of water, which is felt as mechanical stress. Also, clays form a film on the skin's surface which reduces the loss of natural moisture reducing the transepidermal water loss (TEWL), promoting high skin hydration, that can be felt by volunteers as raising in mechanical tension (Berardesca *et al*., 2012). Therefore, it was expected to observe increment in firmness 20 min after mask removal, due to the immediate effect of clay mask on skin, but this was not observed in this study. This time was set with the intention to ensure drying of the skin after removal of the product with distilled water because it could interfere with the skin properties evaluated.

Thus, the composition of the different clays did not influence the skin viscoelasticity behavior in the short-term clinical study. The long-term use of this type of formulation probably would have more benefits because it was already demonstrated that the clay application in rats for 7 days increased the numbers of collagen fibers (Valenti *et al*., 2012), which could decrease wrinkles and increase the skin firmness.

## CONCLUSIONS

The chemical and mineralogical composition of the different clays tested in this study (white, green and pink) did not influence the skin biometric profiles because firmness and elasticity were not affected in a short-term application. Thus, a long-term study is essential to establish the efficacy and mechanism of action of this type of facial mask.

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## RESUMO

*Caracterização e estudo clínico de curta duração de máscara facial argilosa*

**Máscaras faciais argilosas são utilizadas para tratar algumas doenças dermatológicas, apenas para a limpeza ou reduzir a quantidade de óleo secretado pelas glândulas sebáceas. Existem vários tipos de argilas, que variam em composição mineral, química, cor e origem. No entanto, a literatura carece de estudos envolvendo máscaras faciais argilosas, em particular em relação a sua influência nas propriedades biomecânicas da pele. Assim, este trabalho teve como objetivo caracterizar argilas coloridas e avaliar sua influência sobre a firmeza e elasticidade da pele por meio de um estudo clínico de curto prazo. Diferentes argilas (chamadas neste estudo de silicato de alumínio e magnésio - MAS, branca, rosa e verde) foram caracterizadas quimicamente, e formulações de máscaras faciais foram preparadas. O estudo clínico de curto prazo foi realizado por meio da aplicação das formulações na pele. A firmeza e elasticidade da pele foram avaliadas antes do tratamento e após a remoção da máscara. A análise estatística mostrou nenhuma influência significativa do tempo ou das formulações nesses parâmetros, embora os voluntários tenham reportado sensação de tensão mecânica, após a remoção das máscaras faciais argilosas. Assim, a composição das diferentes argilas não afetou o comportamento visco-elástico da pele no estudo clínico de curto prazo, e uma utilização de longa duração poderia ser indicada com a finalidade de se observar todos os benefícios esperados.**

Palavras-chave: Argila. Propriedades biomecânicas. Caracterização química. Máscara facial. Estudo clínico.

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