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Characterization Studies of a Commercial Blue Clay for Cosmetic Textiles with Antibacterial Activity

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ABSTRACT

Since the beginning of the present century, tests have shown that some types of clay can present antibacterial activity. In addition, clays considered for pharmaceutical and cosmetic products have been found to be non-toxic and non-irritating materials, therefore, the use of this types of clays for cosmetic and pharmaceutical purposes has increased in recent years. The above being said, different types of clay have been used over time due to their antibacterial properties, but the analytical methods for their characterization are just beginning to develop. This article is part of a study having as main objective the development of multifunctional antimicrobial textile materials to prevent fungal and bacterial proliferation, thus creating an antimicrobial shield for the human body, especially for blemish-prone skin. In this paperwork, a commercial blue clay was characterized through modern techniques. One of these techniques is X-ray Diffraction (XRD). Coupling SEM with an Energy Dispersive X-Ray detector (EDX), complete information of the morphology and elemental composition of the clay powder can be obtained. Additionally, a microbiological characterization was also performed in order to assess the antifungal properties. Thus, the obtained results provided an overview of the main features of blue clay. Further studies will be directed to the characterization of different types of textile materials, in order to choose a "clay-textile" pair with improved antimicrobial activity.

Keywords: Blue clay, XRD, SEM-EDX, Antimicrobial

INTRODUCTION

Clays are natural materials made from very small particles (< 2 μm), that possess plastic properties, and are usually composed of small mineral fragments consisting of hydrous-layer aluminum silicates. Occasionally, they may also contain magnesium and/or iron.

An application of mineral clays is the treatment of several health conditions such as dermatological diseases, rheumatic inflammations, arthritis and they also can be used in skin moisturizing products (Carretero et al. 2006, Abdel-Motelib et al. 2011, Carretero 2002; Bergaya et al. 2006).

There are some literature studies that confirm that some clays have antibacterial properties. One study shows that silver and quaternary ammonium surfactant-modified clays inhibited the development of E. Coli (Parolo aet al. 2011).

Another paper investigated the antibacterial effect of white, pink, grey and yellow clays against Staphylococcus aureus and Pseudomonas aeruginosa. The results concluded that all four types of clay have an antibacterial effect against S. aureus and the pink mineral clay was the one that had antibacterial effect against P. Aeruginosa (Lafi and Al-Dulaimy, 2011). Regarding the antibacterial effect of blue clay, some studies showed that it has the capacity to inactivate pathogens (Williams et al. 2011; Morrison et al. 2014). A microbiological study performed on blue clay showed that this type of clay does not present a proper environment for the development of Candida albicans, Pseudomona aeruginosa, Staphilacoccus aureus. Thus, blue clay has a high potential for its use in cosmetics and medicine (Rasma et al. 2017).

XRD analysis is considered to be the most suitable method for quantitative analysis for clays, compared to any other technique (Mumme et al. 1996). However, the quantitative analysis remains a major challenge due to the various chemical compositions, preferred orientation, and great structural diversity of clay minerals (Bergaya et al. 2006, Środoń and Środoń 2013, Zhou et al. 2018). Different types of clay have been studied over time by certain instrumental techniques. For example, XRD is an important technique used for elemental or chemical analysis of clays (Newman 1987, Mermut and Cano 2001). Several studies have been carried out using XRD to determine the mineralogical composition of clays and the presence of minerals (Preeti and Singh 2007, Aroke and El-Nafaty 2014).

Scanning electron microscopy coupled with Energy Dispersive X-Ray detector provides results regarding the size and shape of the particles (Sengupta et al. 2008, Chambers et al. 2000).

In this paper, blue clay was characterized using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) coupled with an Energy Dispersive X-Ray detector (EDX). In addition to these, a microbiological characterization was also performed in order to assess the antifungal properties. Taking into consideration all the mentioned aspects, the obtained results provided an overview on the main features of the blue clay.

MATERIAL AND METHODS

Blue Clay

The clay used in the study was purchased from a national supplier and it is known as “Argila Albastra de Raciu” (Raciu Blue Clay).

Scanning Electron Microscopy - Energy Dispersive X-Ray Spectroscopy

Scanning electron microscopy was performed using a Quanta 200 Scanning Electron Microscope (FEI Company). For the elemental analysis of the

samples, an Element EDS System (EDAX-AMETEK), coupled to the electron microscope was used.

Powder X-ray Diffraction

The XRD diffraction analysis of the clay was performed on a Proto AXRD diffractometer, using as X-ray a Cu Kα radiation (λ = 1.54056 Å). The measurement was performed between 2θ angles 5 – 125°. The obtained diffractogram was analysed using Match! Software, and the phase identification was obtained by comparison with the Crystallography Open Database.

Microbiological Characterization

The commercially available blue clay was firstly submitted to bioburden screening, without preliminary processing (sterilization). The clay was inoculated on four semi-synthetic nutritive media, with high specificity towards characteristic growth for various fungi (Malt Agar-Scharlau, Potato Dextrose Agar-Scharlau, Sabouraud Dextrose Agar-Merck, Czapek-Dox-Scharlau) and one semi-synthetic media, for bacterial growth promotion (Yeas Extract Agar-Merck). Each media was sterilized at 121°C, for 115', and then let to cool at ~45°C, then poured in Ø90 sterile Petri plates. A small quantity of blue clay powder was dotted on the surface of each plate without (see Figure 1), and then incubated at 28°C, for 14 days.

Results and Discussions

In Table 1 are presented the SEM micrographs and the EDX graph. Table 2 presents the results of the elemental analysis.

SEM images illustrates that the blue clay appear as small aggregates, while EDX results show a high carbon content. Also, significant percentages of oxygen, silicon and aluminium were identified. Magnesium, potassium, and iron are also found in smaller amounts.

The obtained XRD diffractogram is presented in Figure 1. It can be observed that the sample is highly crystalline, having many diffraction peaks. However, it is exceedingly difficult to perform a correct phase identification,

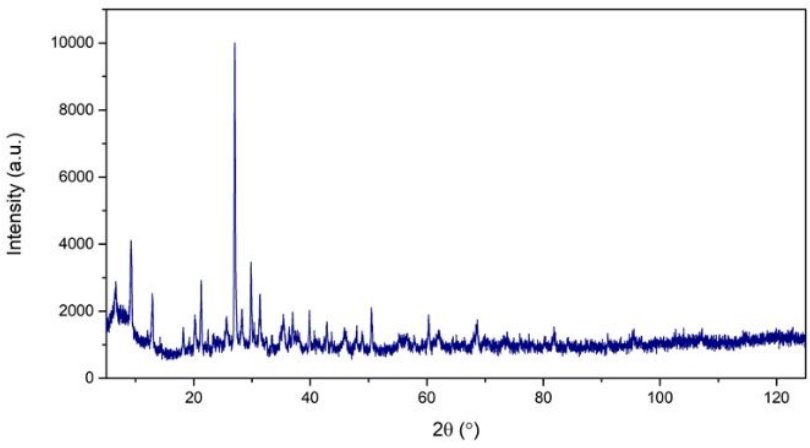
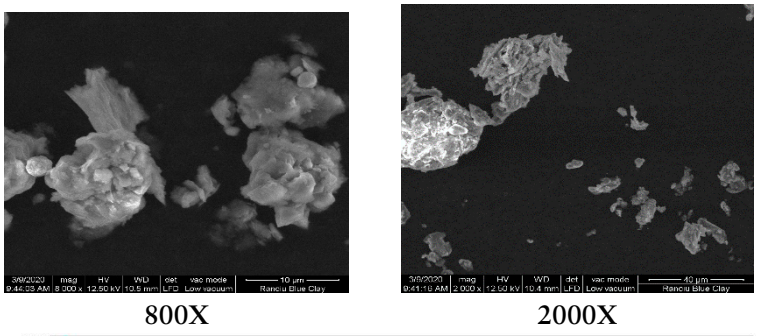


Figure 1: X-ray diffractogram of the clay.

Table 1. SEM and EDX results.



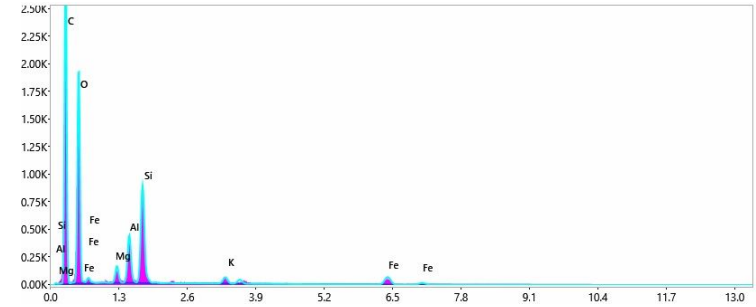


Table 2. EDX elemental analysis.

Element	Amount [%]
C	40
O	27
Mg	3
Al	9
Si	17
K	2
Fe	2

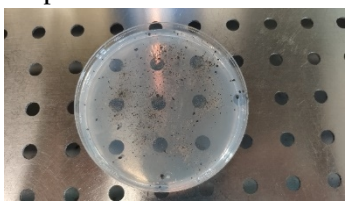
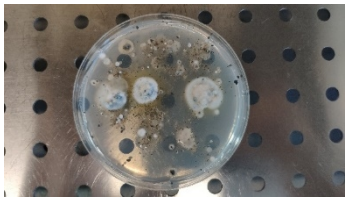
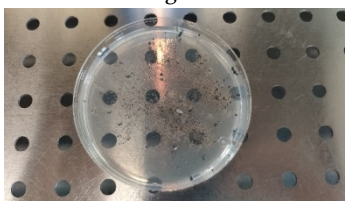
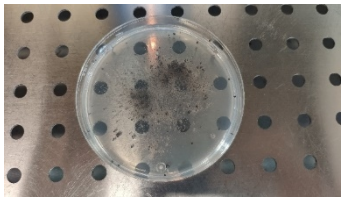
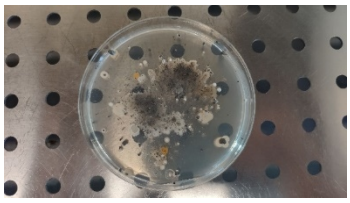
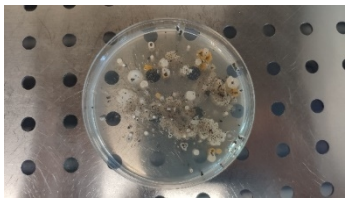
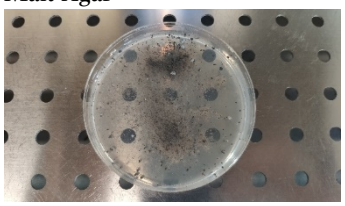
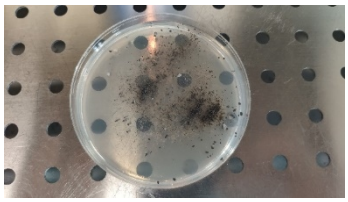
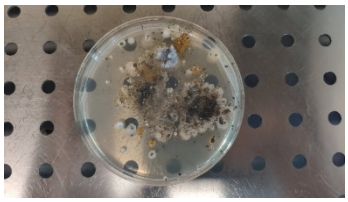
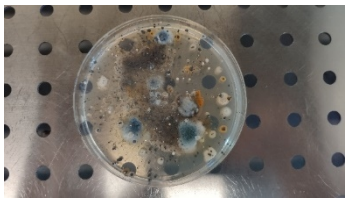
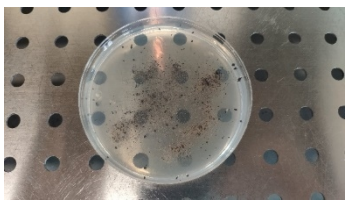
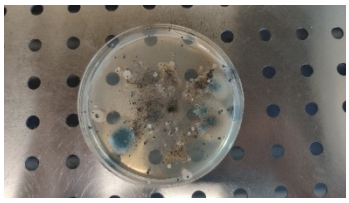
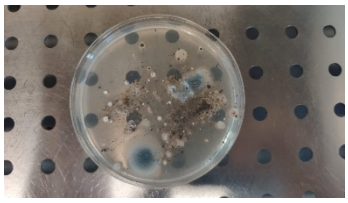
Table 3. Phase composition of the clay.

Entry	Formula Sum	Name	Quantity (%)
96-900-6344	Al_3 Ca $Cl_{1.12}$ $K_{0.99}$ $Na_{2.01}$ $O_{13.76}$ $S_{0.44}$ Si_3	Microsommite	52.6
99-999-9900	Al_2 Ca $H_{3.68}$ $O_{14.13}$ Si_4	Laumontite	24.6
99-999-9904	$Al_{0.58}$ $H_{0.668}$ K $O_{6.25}$ $Si_{1.42}$	Montesommaite	14.9

due to the many different possible components. To perform some identification, additional data should be used, such as elemental composition, obtained from EDX measurements. By choosing the appropriate elements, the possible composition from Table 3 was obtained.

As observed in Table 4, the blue clay powder has a high bioburden load, with several microbial structures, specific to filamentous fungi, yeasts, and bacteria. Frequently, clays can be contaminated during the processing and storing stages (strains of Bacillus, Clostridium etc.) (Bubik 1992). Furthermore, several microbial strains were analysed under an Olympus SZ

Table 4. Bioburden screening plates – initial and after the incubation period.

Initial	After incubation period
Czapek Dox	
	
Yeast Extract Agar	
	
	
Malt Agar	
	
	
Potato Dextrose Agar	
	
	

Continued

Table 4. Continued

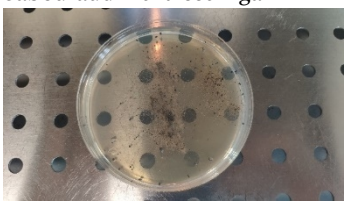
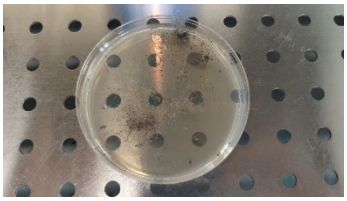
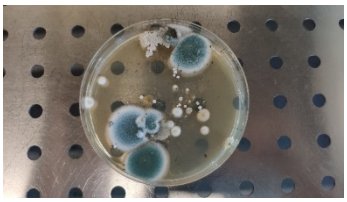
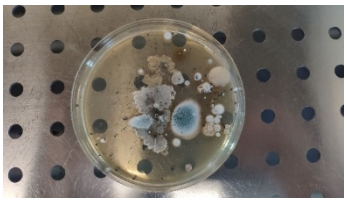
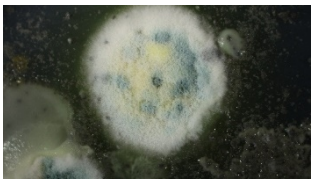
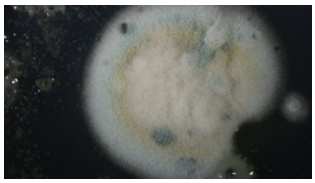
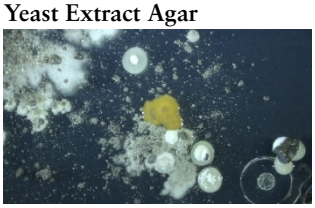
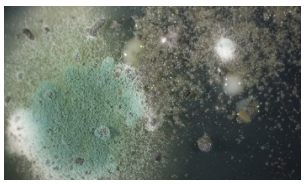
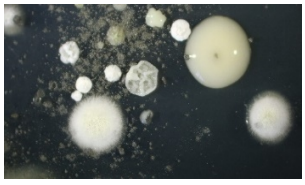
Initial	After incubation period
Sabouraud Dextrose Agar	
	
	

Table 5. Microbial strains stereomicroscopy.

Czapek Dox		
		
Yeast Extract Agar		
		
Malt Agar		
		
Potato Dextrose Agar		
		
Sabouraud Dextrose Agar		
		

61 stereomicroscope (with Greenough Optical System, magnification level 0.67x) for specific morphology assessment (see Table 5).

Microscopy analysis revealed both fungal structures (filamentous and yeast) and bacteria-like structural organizations. Based on specific morphological characteristics, it can be noted the presence of microbial structures belonging to Trichoderma and Penicillium species, although, for a proper identification, molecular assessment of the isolated strains is highly required.

CONCLUSION

Blue clay was characterized in terms of morphological and elemental analysis (SEM-EDX and XRD). Blue clay appears as a small aggregate, containing mostly carbon, oxygen, silica and aluminium and small amounts of magnesium, potassium, and iron. Also, a microbiological investigation was performed. The results obtained for blue clay used in this study are not satisfactory as microscopy analysis revealed the presence of both fungal structures (filamentous and yeast) and bacteria-like structural organizations on the plates. Thus, future studies of this work will be conducted, using a modified blue clay or a different type of clay, as the blue clay powder by itself did not present relevant results regarding microbiological activity.

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