

## Organoclay brazilian smectitic: synthesis and characterization

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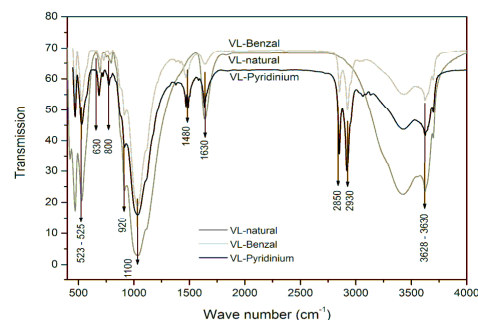
**Abstract** – Organophilic clays have been synthesized from ‘Verde-Lodo’ natural clay from the State of Paraíba (Brazil) using quaternary ammonium salts: cetyl-pyridinium chloride and benzalkonium chloride. The organoclays have been investigated by use of characterization techniques such as thermogravimetric analysis, X-ray diffraction, Fourier transform infrared spectroscopy and N<sub>2</sub> adsorption-desorption (BET method). Thermogravimetric analysis show the appearance of a new decomposition stage: the presence of absorption bands due to the groups CH<sub>2</sub> and CH<sub>3</sub> in the infrared spectra, increase of the basal spacing and decrease of the superficial area of modified clays show consistently successful synthesis of the organoclay.

Organoclays have been widely used for polymeric nanocomposites production improving properties and reducing weight and cost of these new types of materials; in addition organophilic clays present potential for environmental remediation such as adsorption of organic compounds due to the hydrophobic character that obtains by means of the organophilization procedure. In this work, organophilic clay ‘verde-lodo’ bentonite from the State of Paraíba (Brazil) were organofilized by intercalation of two different quaternary ammonium salts. The organoclay were prepared as described by Pereira (2008) [1]. The surfactant used were cetyl-pyridinium chloride (VL-Pyridinium) and benzalkonium chloride (VL-Benzal) in the amount of milliequivalence ratio of 1:1. The obtained material had been characterized by use of X-ray diffraction (XRD), N<sub>2</sub> adsorption-desorption (BET), termogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR). The basal spacing of natural and organophilic clays are showed in Table 1. The clay interlayer space was increased after its modification by surfactants, due to replacement of sodium ions by alkylammonium ions. The clay surface area and micro and mesopores volume decrease intensely after organophilization. This is attributed to the salt cations which may block the interlamellar space and inhibits the passage of N<sub>2</sub>. The termogravimetric analysis shows one first weight loss stage in the temperature range 20 - 100 °C due water loss in all samples. It was higher for natural clay (≈4,5%), which corresponds to the removal of water from the interlayer which is coordinated with Na<sup>+</sup> e Ca<sup>+</sup> ions, while organoclays the weight loss ≈1% lead to the conclusion that alkylammonium ions adsorbed less water. The second decomposition stage appears only modified clays in the temperature range 200 - 300 °C, attributed to the organic matter released of quaternary ammonium salt, and the last loss stage in all samples is attributed to the dehydroxylation of clays. The Figure 1 shows infrared spectra of clays samples in study with absorption bands of the groups CH<sub>2</sub> (2930 e 2850 cm<sup>-1</sup>) and CH<sub>3</sub> (1480 cm<sup>-1</sup>) for modified clays. The results confirm the effectiveness of the synthesis of organophilic clay ‘verde-lodo’ bentonite whose features enable the interaction with organic compounds.

**Table 1:** Structural parameters of natural bentonite and organoclays.

Samples	d <sub>001</sub> (nm)	S <sub>BET</sub> (m <sup>2</sup> /g)	R <sup>2</sup>	V <sub>mi</sub> (cm <sup>3</sup> /g)	V <sub>me</sub> (cm <sup>3</sup> /g)
VL-natural	1.55	65.4144	0.99938	17.024	16.711
VL-Pyridinium	2.14	0.1225	0.99864	0.029	0.027
VL-Benzal	2.76	5.6589	0.99959	1.128	2.514

S<sub>BET</sub>: specific surface area; R<sup>2</sup>: correlation coefficient of BET model;  
V<sub>mi</sub>: volume of micropore; V<sub>me</sub>: volume of mesopore.



**Figure 1:** Infrared spectra of natural clay and surfactant modified clays.

### References

[1] K. R. O. Pereira, Estudo, em escala de laboratório, do uso de argilas do tipo bofe na obtenção de argilas organofílicas e ativadas. 2008. 139p. Tese (Doutorado em Engenharia Química) - Escola Politécnica da Universidade de São Paulo, São Paulo.