

## Synthesis and characterization of an urea functionalized nickel phyllosilicate

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**Abstract** – A urea derived silane was synthesized in order to obtaining a phyllosilicate, through the reaction of the ethylenediamine (EN) with the 3-(triethoxysilyl)propyl isocyanate (ISO). The silane was characterized by CHN elemental analysis, FT-IR spectroscopy and <sup>13</sup>C NMR. A urea functionalized nickel phyllosilicate have been synthesized by sol-gel process. The nickel urea phyllosilicate synthesized by sol-gel process was characterized by CHN elemental analysis, FT-IR spectroscopy, <sup>13</sup>C NMR, <sup>29</sup>Si NMR, X-ray diffraction and surface area.

The immobilization of organic molecules on inorganic surfaces has attracted considerable attention for practical applications. These molecules containing specific groups can change the properties of the inorganic surface as a structural improvement, changes in thermal stability, possible different reactions at the surface, changes in the solubility in organic liquids and water [1]. In this perspective, a urea derived silane was synthesized to obtaining a phyllosilicate, through the reaction of the ethylenediamine (EN) with the 3-(triethoxysilyl)propyl isocyanate (ISO). The white solid product (ISOEN) was obtained by this reaction of two mols of 3-(triethoxysilyl)propyl isocyanate and one mol of ethylenediamine. The route of synthesis is outlined in Figure 1. The formation of the ISOEN was evidenced by the disappearance of the characteristic band of the isocyanate group in 2274 cm<sup>-1</sup>. The Figure 2 shows the FT-IR spectra of the 3-(triethoxysilyl)propyl isocyanate, ethylenediamine and ISOEN. The preparation of phyllosilicate followed the stoichiometric ratio of 1.33 Si / Ni, similar to the natural talc. The Ni<sup>2+</sup> salt was solved in ethanol and then added to an ethanolic solution of ISOEN. To this solution sodium hydroxide was slow added under stirring at 323 K. The solid product was aged for 48 h in the mother liquor. The obtained solid was separated and sequentially washed by deionized water, ethanol and dried in vacuum at room temperature for 6 h. The product (FISOEN-Ni) was characterized by X-ray diffraction showing four peaks, 4.98°, 22.4°, 34.36 and 59.98°. These results suggest the formation of a lamellar structure, similar to the literature results concerning to an organically modified phyllosilicate [1]. The peak at 4.98° corresponds to the interlayer space of 1772 pm. This value is consistent to the organic molecule size in the silane. The peak at 59.98° is associated with interplanar distance of 154 pm, characteristic of the reflection in the 060. The <sup>13</sup>C NMR spectrum of the FISOEN-Ni showed an intense peak at 162.7 ppm, corresponding to the carbonyl carbon. For the carbons of propyl group were assigned the peaks at 44.31 ppm, 23.55 ppm and 9.53 ppm. The two carbons between the urea groups have the same chemical environment and the same sign at 42.28 ppm. These data are agrees with the proposed structure of the organic part of the urea functionalized nickel phyllosilicate, showing the synthesis have not affected the organic part of the silane precursor.

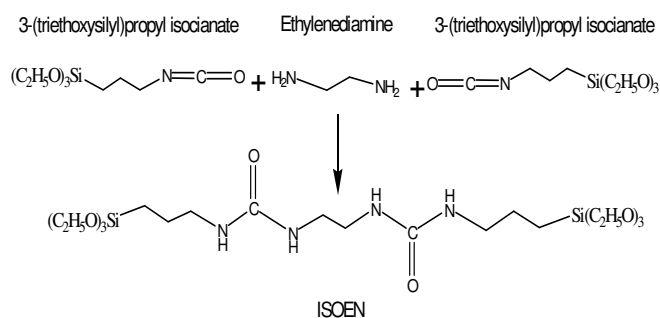


Figure 01: Synthesis scheme for ISOEN

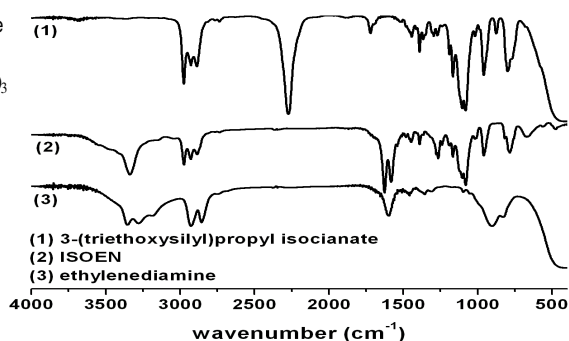


Figure 2: FT-IR spectrum for 3-(triethoxysilyl)propyl isocyanate, ethylenediamine and ISOEN

### References

- [1] M. A. Melo Jr., F.J. V. E. Oliveira, C. Airoldi, Applied Clay Science 42 (2008) 130