

Silica-Supported Proline Derivatives for Catalytic Studies

Tatiane Inforzato¹, Liane Marcia Rossi², Tiago Venancio,¹ Alcindo A. Dos Santos²

¹Departamento de Química, Universidade Federal de São Carlos, Rod. Washington Luis Km 235 – CEP 13565-905, São Carlos – SP. ²Instituto de Química, Universidade de São Paulo, Av. Prof. Lineu Prestes, 748 – CEP 05509-000, São Paulo – SP

*e-mail tatianeinforzato@yahoo.com.br.

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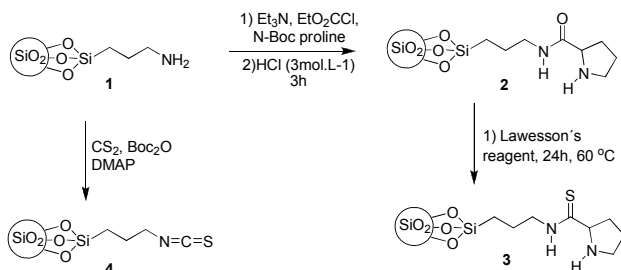
INTRODUCTION

Heterogenization of organic catalysts would be an effective way to improve their catalytic efficiency via catalyst recovery and reuse. Efforts have been devoted to the immobilization and recycling of L-proline¹. In this work, we present the detention of prolinamide and prolinethioamide in silica. These materials can be applied in catalytic reactions.

RESULTS AND DISCUSSION

Initially, for the functionalization of the silica surface with proline we prepared compound **1** which was treated with ethyl chloroformate followed by N-Boc-proline to afford **2** according Scheme 1. The Boc group was removed by acid treatment, allowing the preparation of the hybrid organo-inorganic material **2**. All intermediates as well the target material **2**, were submitted to characterization analysis, including ¹³C-NMR/CPMAS. Material **2** was also submitted to microanalysis and was found that the functionalization resulted in 0.46 mmol of organic residue/g of silica. Compound **2** was converted into the corresponding thioamide by treatment with Lawesson's reagent. Additionally, compound **1** was converted into **4** which will be submitted to reaction with prolinamide as well prolinethioamide to prepare the corresponding thioureas.

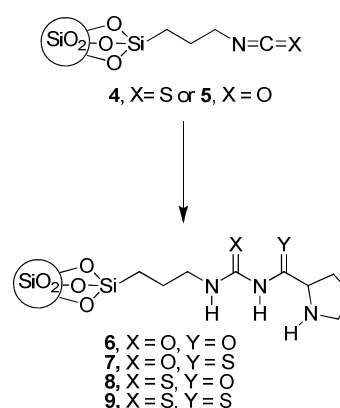
Scheme 1. Funcionalization of the prolinamide and prolinethioamide.



Materials **1-4** were submitted to IR, ¹³C-NMR/CPMAS, ²⁹Si-NMR and microanalysis and are in agreement with the proposed structures.

Figure 1 presents the designed organo-inorganic hybrid materials that will be prepared from **4** or **5**.

Figure 1. Ureas and Thioureas that will be prepared



Next, these materials will be tested as catalysts for Michael addition, Strecker and Mannich reactions.

CONCLUSION

In conclusion, we demonstrated the preparation of hybrid organo-inorganic materials by efficient procedures. The new material **3** was produced to be used as catalyst in order to investigate the performance compared with **2**. Tests catalysis using this material is already being carried out in our laboratory.

ACKNOWLEDGEMENTS

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REFERENCES

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