

Development of nano oxide α -CoMoO₄ by soft chemistry

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Abstract. Molybdates oxides as AMoO₄ (A = Cu, Co), have remarkable properties. These properties depend strongly on the crystallite size. Nanostructured Powders of these molybdates make their applications more usable.

The aim of our work is to synthesize oxide CoMoO₄ by soft chemistry method, which has the advantage of producing very fine and homogeneous powders, which increases their reactivity. The products obtained were characterized by XRD, SEM and TEM.

1. INTRODUCTION

Control the morphological characteristics of nanoscale particles of materials is of great interest to optimize their properties in a given application domain [1,2], or for carrying out type materials to facilitate the comparison with theoretical models.

Several synthetic methods both physical and chemical have been developed to obtain materials with well-defined morphological characteristics. Physical methods [3–6] are based on the decomposition of a solid material in order to reduce the size. These methods generally require complex and costly installations without necessarily ensure the achievement of homogeneous particles. On the other hand chemical methods [7–10], are less expensive, more affordable and consist of particles grow using molecules as departure entities. The soft chemistry provides particularly materials while controlling the morphology, grain size and stoichiometry. This has already been verified in developing of CuMoO₄ [11, 12].

The objective of our work is to use the soft chemistry which has the advantage of producing a very fine powders and homogeneity so as to develop a nanoscale powder of CoMoO₄.

2. EXPERIMENTAL METHODS

The reagents used for the synthesis are: ammonium heptamolybdate (NH₄)₆Mo₇O₂₄ · 3H₂O (Acros), the salt metal Co(NO₃)₂ · 6H₂O (Aldrich), citric acid (CA) (Aldrich), ammonia (NH₄OH) 28% pure, density = 0.91 and nitric acid (HNO₃ 68%, density = 1.83).

The synthesis of CoMoO₄ oxide powders is realized by dissolving in an aqueous the nitrate cobalt in the presence of ammonium molybdate in stoichiometric amount (1/1). To this mixture is added an excess of citric acid (acid / Cations = 3), the pH of the solution is set at 3. Evaporation of the resulting solution at 80 °C leads to the formation of a gel noted G. The Treating the precursor obtained after precalcination in air at 300 °C, allows to obtain molybdate CoMoO₄. The synthesis protocol is summarized in Figure 1.

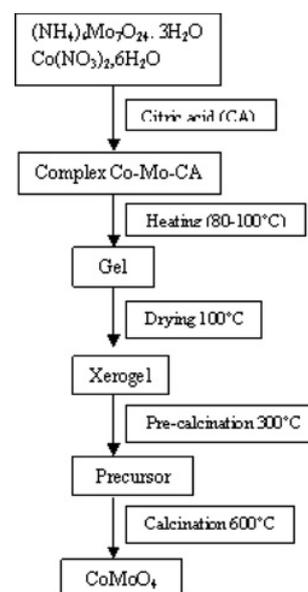


Figure 1. Scheme of CoMoO₄ synthesis.

3. RESULTS AND DISCUSSION

3.1. Thermal behavior of xerogel obtained at 100 °C

Thermogravimetric analysis of xerogel obtained by drying at 100 °C the G gel was carried out in air with a temperature rise of 2.5 °C / min. The curve obtained (Figure 2) shows a decomposition in several stages. The total mass loss of xerogel citrate is about 60%. This loss can be attributed to deaeration, the departure of ammonia molecules and the combustion organic species. The final temperature of decomposition is about 600 °C. After no phenomenon is observed, indicating that the decomposition is complete and the oxide CoMoO₄ is formed.

3.2. Analysis by X-ray diffraction of the precalcined and calcined xerogel

The X-ray diffraction diagrams show that treatment of the xerogel at 300 °C leads to an amorphous phase and the

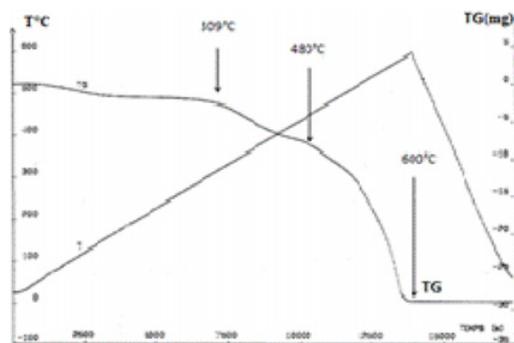


Figure 2. Thermogravimetric analysis of xerogel at 100 °C.

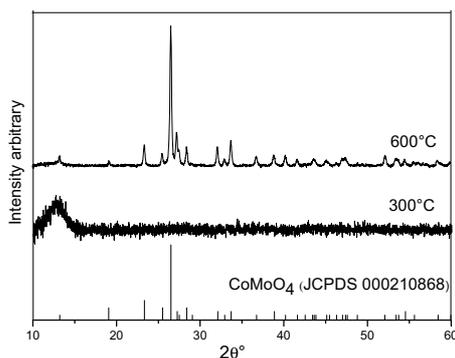


Figure 3. X-ray diffraction of the cobalt xerogel calcined at 300 °C and 600 °C.

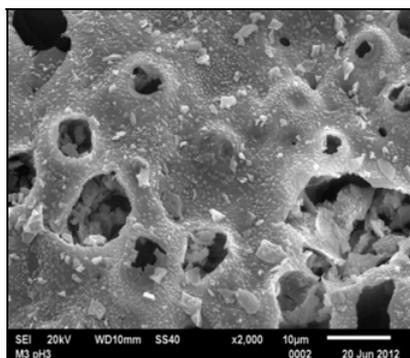


Figure 4. SEM micrograph of the compound αCoMoO_4 .

heat treatment at 600 °C for 2h gives the pure molybdate αCoMoO_4 (JCPDS 000 210 868) (Figure 3).

3.3. Analysis by electron microscopy of molybdate oxide αCoMoO_4

The αCoMoO_4 oxide, prepared by heat treatment of xerogel G was examined by electron microscopy.

The SEM image of the αCoMoO_4 phase (Figure 4) shows that the powder is formed of porous agglomerates forming cages. The porosity observed comes from rapid gassing during the decomposition of xerogel.

The micrograph TEM (Figure 5) shows that the particles are well dispersed, with more or less spherical shapes and sizes nanoscale estimated at about 60 nm. This value is of the same order of magnitude as that calculated by the Scherrer formula (≈ 70 nm).

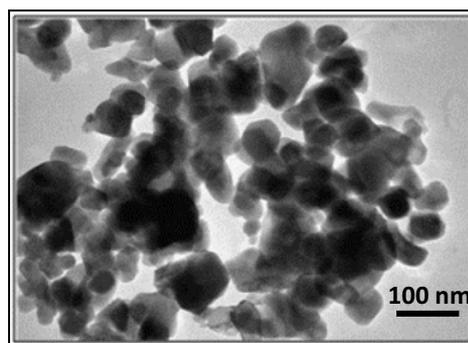


Figure 5. TEM micrograph of the compound αCoMoO_4 .

4. CONCLUSION

In this work, we have developed molybdate αCoMoO_4 by sol-gel from ammonium heptamolybdate, the metal precursor $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and a complexing agent (citric acid) at low temperature. The final calcination temperatures were determined by TGA-DTA. The purity of the obtained phases was confirmed by X-ray diffraction; their morphology was examined by SEM and TEM.

The TEM micrographs of Molybdate, show that the particles are well dispersed and their nanoscale size is estimated to be about 60 nm.

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References

- [1] H. Ehrenberg and H. Weitzel. *Phys. Rev. B: condens mater. Phys.*, **61**, 16497–1650 (2000).
- [2] M. Gaudon, A. E. Thiry, A. Largeteau, P. Deniard, S. Jobic, J. Majimel, and A. Demourgues, *Inorg. Chem.*, **47**, 2404–2410 (2008).
- [3] H. Zeng, W. Cai, Y. Li, J. Hu, P. Liu, *J. Phys. Chem. B*, **109** 18260 (2005).
- [4] N-H. Duc, T-M. Danh, N-A. Tuan, J. Teillet, *Appl. Phys. Lett.*, **78**, 3648 (2001).
- [5] S. Wagner, J-L. Shay, B. Tell, H-M. Kasper, *Appl. Phys. Lett.*, **22**, 351 (1973).
- [6] H. Garcia-Miquel, S-M. Bhagat, S-E. Lofland, G-V. Kurlyandskaya, A-V. Svalov, *J. Appl. Phys.*, **94**, 1868 (2003).
- [7] K. Das, S-K. Panda, S. Gorai, P. Mishra, S. Chaudhuri, *Mater. Res. Bull.*, **43**, 2742 (2008).
- [8] L-A. Palacio, A. Echavarri, L. Sierra, E-A. Lombardo, *Catalysis Today*, **107**, 338 (2005).
- [9] J-L. Bates, L-A. Chick, W-L. Weber, *Solid State Ionics.*, **52**, 235 (1992).
- [10] M-D-B. Arnes, A. Menta, T. Thundat, R-N. bhargava, C. Chhabra, B. Kulkarni, *J. Phys. Chem.*, **104**, 6099 (2000).
- [11] J. Zhang, Z. Zhang, Z. Tang, Y. Lin, Z. Zheng, *J. Mater. Proc. Techn.*, **5622** (2002) 1.
- [12] M. Benchikhi, R. El Ouatib, S. Guillemet Fritsch, J-Y Chane-Ching, L. Er-Rakho, B. Durand. *Materials Letters*, (to be published).