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# Synthesis, characterization and catalytic properties of ternary hybrid material based on cetyltrimethylammonium bromide and n-hexadecylamine in $WO_3 \cdot H_2O$

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**Abstract** – New hybrid composites based on $WO_3.nH_2O$ containing intercalated cetyltrimethylammonium bromide and n-hexadecylamine were prepared. Catalytical studies showed that the presence of both surfactants in the structured of $WO_3$ (ternary hybrid) leads to a material with interesting catalytic effect on hydrocarbon oxidation.

The combination of organic and inorganic components can lead to a material with unusual properties that might not be achieved with each component separately. Construction of these networks can be tailored by the adequate use of sol-gel method, enabling one to tune the hydrolysis/condensation of the metallic cations with the self-assembly of the organic counterparts. Among several metal oxides, tungsten oxides are used in catalysis, display devices, or sensors. Accordingly, efforts have been devoted to designing novel methods for the synthesis of materials with different characteristics. In this context, we report the synthesis of a new ternary hybrid tungsten oxide via surfactant templated soft chemistry approach. Further, the catalytic activity of this material toward the oxidation of cyclooctene and styrene in the liquid-phase was investigated.

Figure 1 depicts the XRD patterns of the $WO_3.nH_2O$ xerogel (Figure 1a) and of the ternary hybrid material cetyltrimethylammonium bromide/n-hexadecylamine/tungsten oxide hydrate ($WO_3$/CTAB/HDA) (Figure 1b). For the hybrid material there are three main peaks at 2θ 2.45°, 3.22°, and 6.20°, exhibiting a *d*-spacing approximately 3.60 nm, and the pattern is in according with a bi-dimensional structure.

The scanning electron microscopy images of $WO_3$/CTAB/HAD (Figure 2) show a bi-dimensional structure throughout the sample originated from the region previously occupied by surfactants in the interlamellar domain, *i.e*., the material exhibits a lamellar arrangement, corroborating the diffraction results. In addition, the images show the presence sheets consisted of oxide layers with nearly 150 μm of thickness.

In order to investigate the catalytic activities of $WO_3$ ternary hybrid material, we proposed oxidation of three hydrocarbons: (z) cyclooctene, styrene and cyclohexane, employing $H_2O_2$, tert-butyl hydroperoxide and m-chlorperbenzoic acid as oxygen transfer agents. The catalytical oxidation results were calculated based on percentage yields. Our results are very satisfactory (concerning cyclooctene epoxidation, the yield found was 80%) when compared with those reported yields for cyclooctene epoxidation reactions catalyzed by vanadium pentoxide supported on silica, even with porphyrinic complexes. For oxidation of cyclohexane, best results were achieved by the use of t-BOOH as oxidant, leading to 100 % conversion, at both 25 and 60ºC temperatures, selectively to cyclohexanone.

Overall, it is possible to consider that the preparation of ternary composites can become an effective approach toward the improvement of the functional performance of this class of hybrid materials. Moreover, the synthetic route adopted herein is a simple and low-cost alternative for the preparation of ternary hybrid materials for catalytic purposes.

**Table 1:** Products yields (%) obtained by the oxidation of cyclooctene, cyclohexane and styrene respectively, using $H_2O_2$, t-BOOH and *m*-CPBA as oxidant and $WO_3$/CTAB/HDA as catalyst at 60°C.

| Entry | Oxidant | Cyclooctene oxide yield (%) | Cyclohexanone yield (%) | Benzaldehyde yield (%) |
|---|---|---|---|---|
| 1 | $H_2O_2$ | 11 | 26 | 2 |
| 2 | t-BOOH | 70 | 100 | 100 |
| 3 | m-CPBA | 80 | - | 6 |

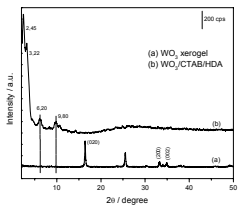
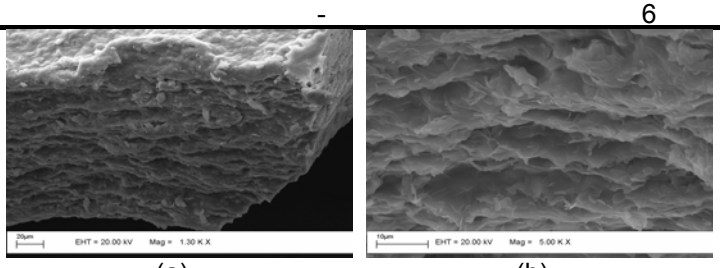



**Figure 1:** DRX **a)** $WO_3$ xerogel and **b)** $WO_3$/CTAB/HDA




(a) (b)

**Figure 2:** SEM image of **a)** $WO_3$/CTAB/HDA 1.3KX and **b)** $WO_3$/CTAB/HDA 5KX

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