# UREA FORMALDEHYDE COATED WITH SILICA (NANO) CAPSULES SYNTHESIS IN ULTRASONIC FIELD

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## ABSTRACT

Silica nanomaterials with meso- and macroporosity are of great interest due to their variety of potential applications. These hybrid nanomaterials, including hollow silica nano and micro-particles have attracted more and more attention with potential applications in medicine, environmental protection and analytical chemistry. In this work we present a fast and simple method for of urea-formaldehyde (UF) nanoparticles obtained in basic medium encapsulation by a thin shell coating of silica, thus yielding organic–inorganic hybrid materials. It was employed (U:F): $SiO_2$: mole ratios of (1:1):5; (1:1):10; respectively. The influence of reaction parameters variation as temperature, pH, and time were studied. The morphology and structure of the products were characterized by FTIR spectroscopy, and Scanning Electron Microscopy; SEM. It was put in evidence some of resulted samples characteristics that are correlated to synthesis conditions.

## INTRODUCTION

Organic–inorganic hybrid materials composed of organic polymers and silica constitute an important class of advanced composites, including phenol-formaldehyde resin [1]. These hybrid nanomaterials, including hollow silica nano and microparticles have attracted more and more attention with potential applications in medicine, environmental protection and analytical chemistry. One important advantage of this technique is its versatility to obtain homogeneous organic–inorganic hybrid composites with different structural characteristics and morphology [2-3].

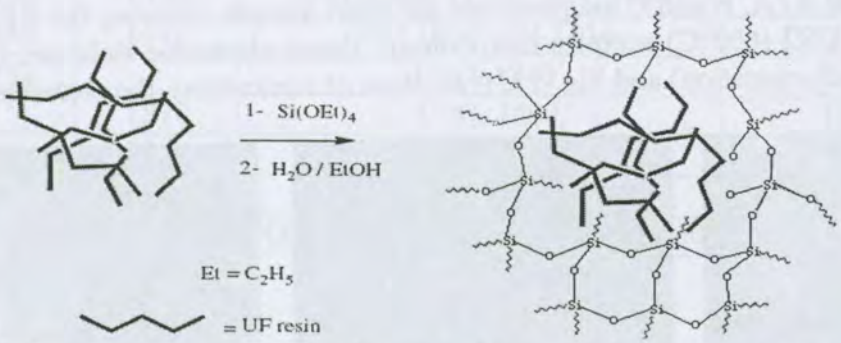



Figure 1: Schematic representation of encapsulated nano-micro-sized UF particles inside silica, hybrid material [3]

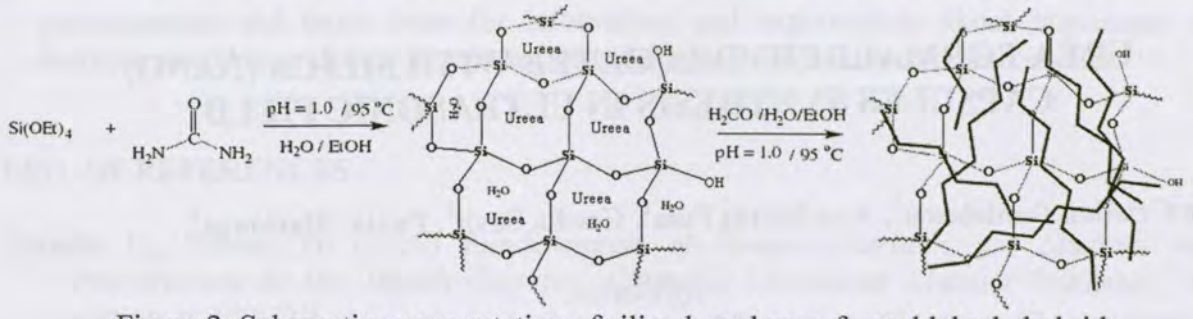



Figure 2: Schematic representation of silica-based urea-formaldehyde hybrid nanocomposites [3]

## MATERIALS and METHODS

It was prepared a series of silica - (urea-formaldehyde) nanocomposite samples, following sol gel synthesis procedure.

$$\equiv Si\text{-}OR + H_2O \rightarrow \equiv Si\text{-}OH + R\text{-}OH \quad (1);$$

$$\equiv Si\text{-}OH + HO\text{-}Si\equiv \rightarrow \equiv Si\text{-}O\text{-}Si\equiv + H_2O \quad (2);$$

It was employed (U:F)-TEOS: molar ratios of - (1:1)-5, (1:1)-10, respectively

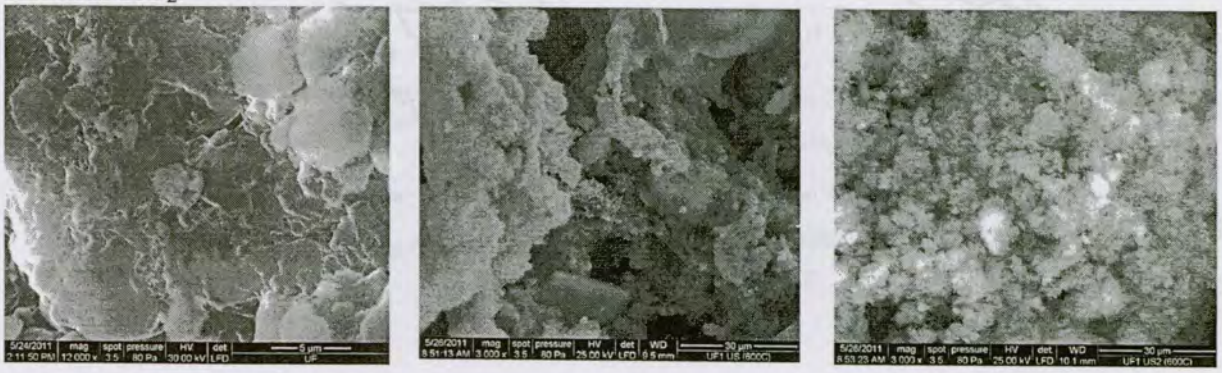
Materials: Tetraethoxysilane (98%, Merck-Germaine); Urea (Fluka-Germaine); Formaldehyde (37%, Viromet-Romania); Distilled water; Sodium hydroxide (10%, Chemapol-Czech Republic) Hydrochloric acid (35%, Merck-Germaine), Formic acid (Silal-Romania).

UF Synthesis: -20 g of urea and 27.2g Formaldehyde (37wt %) were mixed in 150 ml round glass. The pH of solution was adjusted to 7-8 with sodium hydroxide (0.3 ml) after the urea dissolved. During vigorous stirring (350 rpm.) the obtained mixture was corrected to an acid pH 3-4, with formic acid (0.06 mL). The temperature was kept at 90°C for 1 hour. Finally obtained mixture was brought to pH 7-8 (NaOH).

UF particle coating in ultrasonic field with $SiO_2$: - 4 g powder UF was mixed slowly with 100 ml distilled water acidified by hydrochloric acid (pH-1.5), and 10 mL TEOS in the 150 mL bottle glass. Sonication was applied for 40, 47 min. at the 40°C for sample UF1_US (one step of sonication), and for sample UF1_US2 (two steps of sonication). Once the reaction was completed, the particles were gathered by centrifuging and were washed repeatedly with water.

## RESULTS and DISCUTIONS

In the Figure 3., A, B and C are presented the SEM images, showing the UF, UF1_US (600°C) and UF1_US2 (600°C) samples morphology. Using ultrasonic field can be seen in Fig. 3 B (one step of sonication) and Fig. 3 C (two steps of sonication), the coated particles of UF with $SiO_2$.



*a) UF* *B) UF1_US* *C) UF2_US*

Figure 3. SEM images of the UF, UF1_US (600°C/6h) and UF1_US2 (600°C/6h) samples

Ultrasonic field

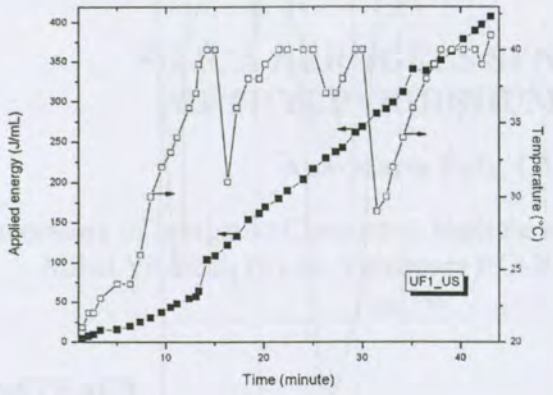



Figure 4. Variation of ultrasonic energy and temperature, applied to the system during synthesis for UF1-US sample

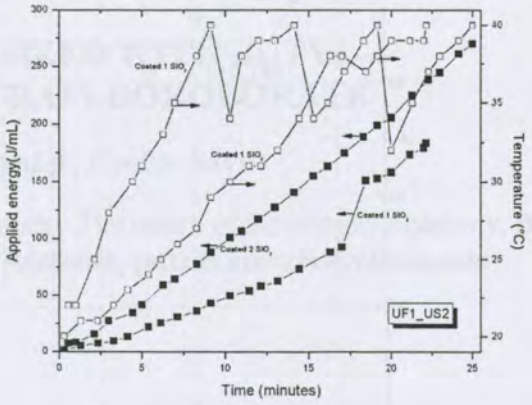



Figure 5. Variation of ultrasonic energy and temperature, applied to the system during synthesis for UF1-US2 sample

FTIR spectra

All obtained sonogels (derived materials fired at different temperatures) were analyzed by FTIR spectroscopy. We have noticed the mean 2 groups of characteristic signals (Table 1): features which are due to urea-formaldehyde resin UF, found at 3419 cm-1 and 3501 $cm^{-1}$; [4], Si-O-Si polymeric network: 1090 $cm^{-1}$, 806 $cm^{-1}$, C=O, 1513-1870 $cm^{-1}$, -OH, 806 $cm^{-1}$ [5-7]. In all FTIR spectra of synthesized samples characteristic bands of these two groups are present.

Table 1. FTIR characterization of samples UF1_US and UF1_US2

| **UF1_US 600°C** | **UF1_US2 600°C** | **Atribution ($cm^{-1}$)** | **REFERENCES** |
|---|---|---|---|
| 463 | 467 | C-Br ,690-515 | [8] |
| 673 | - | C-Br ,690-515 | [8] |
| 800 | 806 | -OH, 750; Si-O-Si, 810 | [10]; [11] |
| 1090 | 1097 | Si-OH, 1100;-C-O; Si-O-Si, 1090; -OH, 1500; | [10]; [11] |
| 1490 | 1513 | C=O, 1508; - C-N, -NH, 1544 | [12]; [13] |
| 1630 | 1630 | C=O,1650;-NH 1634 | [11]; [12] |
| 1780 | - | -C=O, 1740 | [11] |
| | 1870 | - C=O, 1800 | [8] |
| | 2811 | -CH= 2690-2884 | [9] |
| 2879 | 2882 | -C–H, 2958; | [13]; |
| 3501 | 3419 | -OH, 3100-3600;- $NH_2$, -NH -3400 | [10]; [14]. |

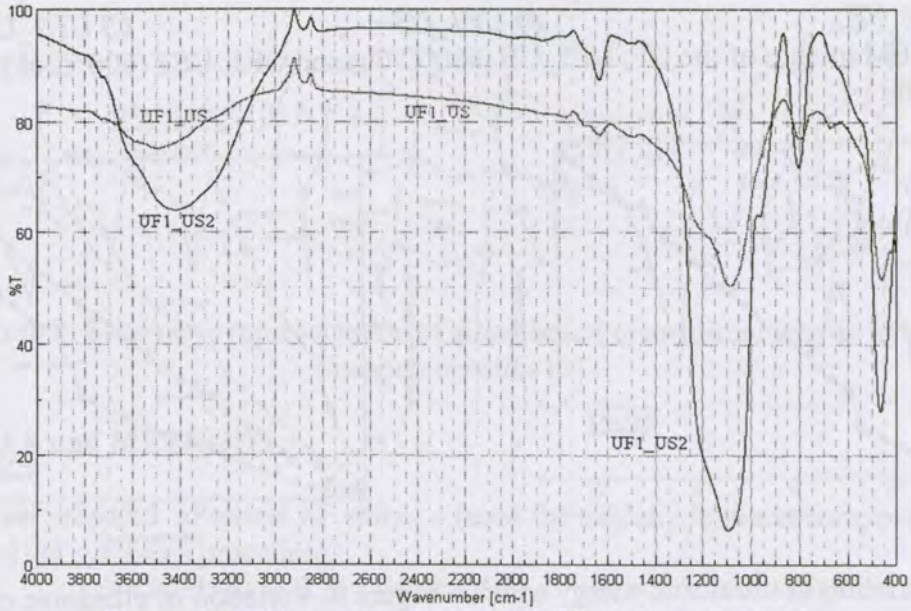



Figure 6. FTIR spectra 1 (U:F)-TEOS (1:1)-5; (1:1)-10

## CONCLUSIONS

In this paper we tried to demonstrate that a one step and two-step sonogel process can be used to treat and redisperse nanopowders to generate a thin layer of silica coated on the particle surface. The application of power ultrasound fulfils two functions: it initiates and sustains reactions leading to the growth of silica and disperses the nanoparticles in solution. As a result, the coated powders were more stable when dispersed in aqueous media. This process offers an alternative route for coating nanopowders used in various technological applications. Morphologic and texture properties measuring are in progress.

## AKNOWLEDGMENTS

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