MICROWAVES-ASSISTED CHEMICAL SYNTHESIS OF SILVER PHOSPHATE POWDER, ITS MORPHOLOGICAL CHANGE AND PHOTOCATALYTIC ACTIVITY WITH SUNLIGHT

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Abstract. Three types of materials in silver phosphate powder were obtained by means of the microwaves-assisted chemical synthesis method. Each type of material in powder have a particle size and morphology characteristic, according has been subjected additional to thermal or surfactant treatment. The surfactant employed is sodium dodecyl sulfate and the treatment temperature was 200 ^{O}C. The material in powder obtained without any additional treatment, have size particle in micrometers and polyhedral morphology, while with the surfactant treatment the size particle is nano metric and its pseudo spherical morphology. The thermal treatment gives origin to powder particles with size micrometric and sub micrometric, buth with morphology of polyhedrons with shape corners and edges. The photocatalytic activity of these three materials is measure under sunlight to degradation of methylene blue in 10 ppm. The type of silver phosphate powder more efficient are the obtained by microwaves-assisted chemical synthesis with additional thermal treatment.

Key words: *Polyhedral morphology, methylene blue, photoelectric effect.*

INTRODUCTION

Heterogeneous photo catalysis is a promising approach for environmental technology improving biological degradation due to high toxicity, a biggest XXI century problem. Today, powder material has been demonstrated an easily-made and economical method to obtain carbon-doped amorphous TiO_2 that is efficiency for the chains (rhodamine B) degradation under visible light from this photo catalyst [1]. The chains degradation using this powder material is reached through a mechanism of water splitting accompanied by an adsorption process, which takes place in in the presence of sunlight. Nevertheless, employing other semiconductor type in powder could be improved in the reduction of photo degradation time of organic chains. In research recent [2-5] has been showed the obtaining of silver phosphate (Ag_3PO_4) by different variants of chemical reduction synthesis and its efficient of this powder to the degradation methylene blue and rhodamine B. The improvement photocatalytic activity of Ag_3PO_4 powder is due to the following underlying mechanism of splitting water: it is well known that any semiconductor exhibit the property known as photoelectric effect that makes them to absorb photons and produce electrons owing to their intrinsic bandgap structure. Even more, the morphology of the particles from semiconductor powder, favors the valence electrons (photo carriers) transports to its surface, which has been demonstrated when the Ag_3PO_4 particles have tetrahedral and cubic morphologies [6]. This counterintuitive behavior is well known in recent studies using the same semiconductor [3, 7] and Ag-Ag_3PO_4 hetero structures powders [4, 5, 8] where to a low surface adsorption area have a high photocatalytic activity for the Rhodamine B degradation. Although, the contact of dye molecules with Ag_3PO_4 implies the photocatalytic gradual deterioration as has been reported in elsewhere [7], as well improve of this aspect [9]. Therefore, in this research is evaluated the photocatalytic activity of materials in Ag_3PO_4 powder under sunlight to degradation of methylene blue. These powders are obtained by microwaves-assisted chemical synthesis method and thermal or surfactant treatment.

MATERIALS AND METHODS

Chemical reagents: All the chemical reagents used were of analytical grade. Ammonium acid phosphate (NH4H2PO4, from JT Baker brand), Ammonium hydroxide (NH4OH from JT Baker brand), Silver nitrate (AgNO3, from JT Baker brand), methylene blue ($C_{16}H_{18}N_3CIS$ from JT Baker brand), the surfactant sodium dodecyl sulfate (SDS from HYCEL) and ultra-pure deionized water (also from JT Baker brand).

Preparation of Ag_3PO_4: First procedure. Two solutions were prepared, one for silver nitrate (Solution A) and another for acid phosphate for ammonium (Solution B), in concentrations of 0.15 and 0.10 respectively, after which solution B was added to the solution A and remained in constant turmoil. The pH = 9 was adjusted with the help of ammonium hydroxide by drip of the same. The final solution was brought to microwave exposure for a time of 7 minutes. The solution obtained was centrifuged and washed with distilled water, the product in powder was decanted and dried for a period of 30 minutes. Second procedure. To obtain powders with nano metric particle size, after mixing solutions A and B, the surfactant SDS was added to 1% by weight of the total mixture, then proceed as in the first procedure. Finally in a *third procedure*, the Ag_3PO_4 powder that is obtained as in the first procedure was subjected to a heat treatment of 200°C for a time of 30 min.

Photocatalytic activity evaluation: In a typical solution of methylene blue (MB) at a concentration of 10 ppm, was brought to sunlight exposure in a batch reactor with air pumping and with a ratio of 200 mg of catalyst per liter of solution.

Characterization methods: The characterizations of the materials in Ag_3PO_4 powder obtained were made by Electron Microscopy of Dispersion (SEM), X-Ray Diffraction (XDR), UV Spectroscopy (ERDT).

RESULTS

The X−ray diffraction pattern obtained is shown in the image left of figure 1. The diffraction planes describe the Ag3PO4 unit cell. The crystalline structure of Ag_3PO_4 synthesized in a basic medium exhibits the characteristic diffraction peaks of this material in angles of 20.9, 29.8, 33.4, 36,7, 42.7, 52,9, 55,0, 57.5, 61,9 and 63.9 degrees attributed to diffraction planes (110), (200), (210), (211), (220), (310), (222), (320), (321), (400), (330) and (411), respectively according to the JCPDS tab 06-0505. The FWHM was calculated with least-squares fit 0.06 degrees in equipment precision value. Also, the synthesis optimal time is to 7 min. because to 5 min. the crystallinity formation starts, while to the 10 min. it is destroyed.

In figure 2 is showed the morphologies obtained by three types of materials in Ag_3PO_4 powder. Figures 2a) and 2b) shows the particle polyhedral morphology with micrometrical size of Ag_3PO_4 powder without any treatment, while the effect of heath treatment decreases the particles size to sub micrometrical scale and change the morphology to polyhedrons with shape corners and edges more pronounced, as can be seen in Figures 2c) and 2d). Conversely, when the particles of this semiconductor are treatment with the surfactant SDS, the morphology is very simple: the particle conglomerates are pseudo-spheroids of Nano metric and sub micrometric size (Fig. 2e and 2f).

In the figure 3 are shown the results of photocatalytic activity evaluation, which can be observed the degradation aliquots of the methylene blue dye in a concentration of 10 ppm for the $Ag_3(PO_4)$ powder with thermal treatment. During the 35-minute span I reached a degradation of 41.55%. This taking into account that no type of lamp was taken, it was simply done with the help of sunlight. For the case of $Ag_3(PO_4)$ powder with the surfactant SDS treatment, the degradation is slightly smaller than the case previously said. While for the case of $Ag_3(PO_4)$ powder without any treatment, the photocatalytic activity is slightly older. All these results can be confirmed by means of dye MB concentration degradation curve, which can be seen in the figure 1b). An advantage that have the $Ag_3(PO_4)$ powder with thermal treatment is their stability improvement as has been reported in elsewhere [9].

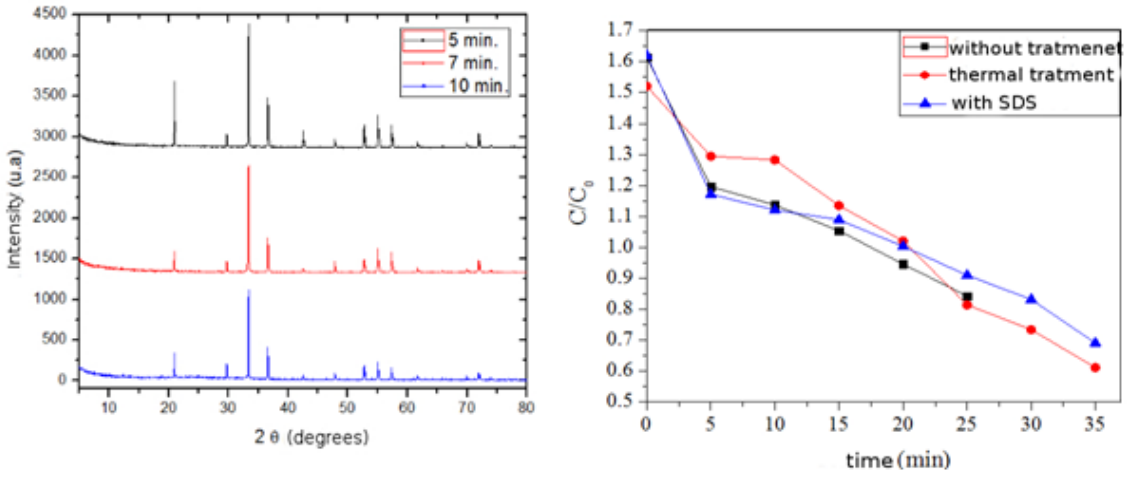

Figure 1. Left image represent the Ag3PO4 DRX diffraction in basic medium showing diffraction peaks. Right image represent a comparative graph of dye MB concentration degradation curve

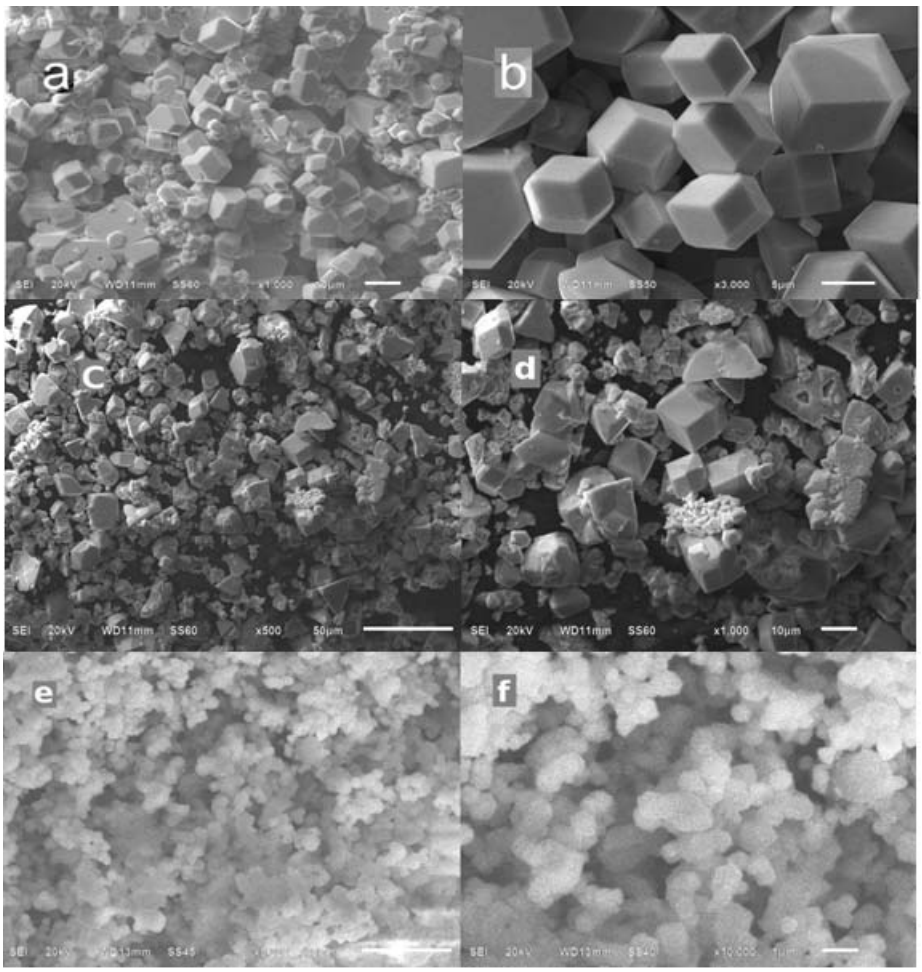

Figure 2. Images of Ag_3PO_4 powder obtained by SEM characterization. a) micrometric particles size and polyhedral morphology to 1000 X without any treatment, b) the image is zoom of a) to 3000 X, c) micrometric and sub micrometric particles size and polyhedral morphology with shape corners and edges to 500 X with thermal treatment, d) the image is zoom of c) to 1000 X, e) particles conglomerates with pseudo-spherical morphology of Nano metric size to 5000 X with surfactant SDS, f) the image is zoom of e) to 10000 X

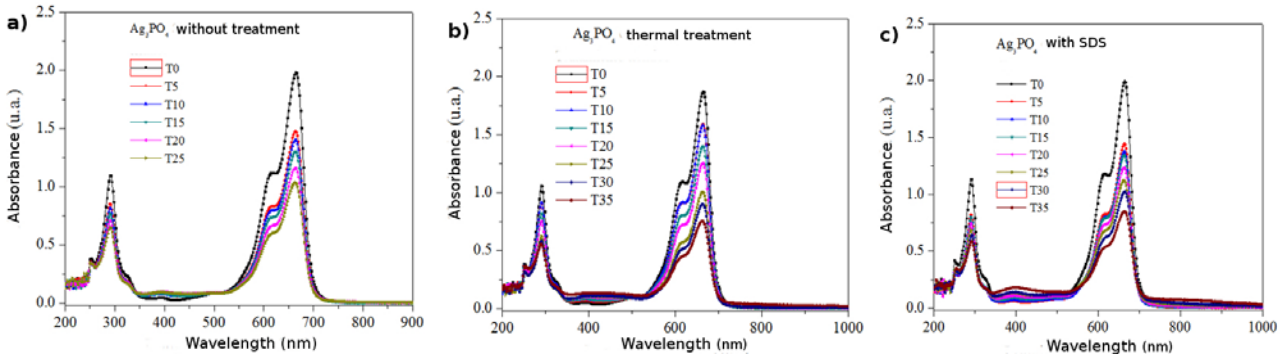

Figure 3. UV spectrum taken from degradation of $Ag_3(PO_4)$

CONCLUSIONS

According to the results obtained, it is concluded that the size and the modification of the morphology of the particles of our three types of samples influence the photocatalytic activity.

The best result to perform the photo catalysis was with the sample to which a heat treatment was applied due to the observations of the vitas morphology in the Scanning Electron Microscope.

In combination with Nano metric and sub micrometric size of polyhedral morphology with good results are obtained to degrade the blue methylene dye.

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