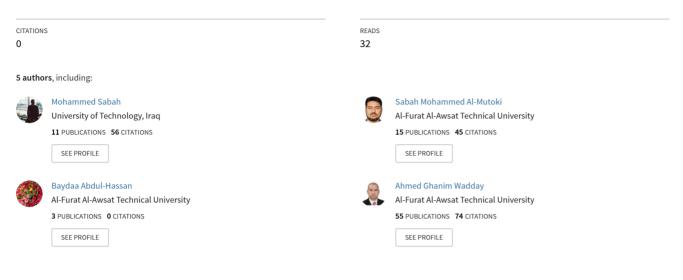
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Room Temperature Grown ZnO Nano Rods Produced by PDC Technique for Drug Industry

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Abstract

In this research we succeed to produce ZnO nano rods which suitable to use for drug industry especially in the field of dermolotical drugs at room temperature by polymer derived ceramic technique (PDC). X-ray diffraction (XRD), and scanning electronic microscope (SEM) were used to detect the critical concentration of zinc sulfate hydro acetate alkoxide used to precipitate ZnO nano rods at room temperature, and they were also used to detect morphology of the produced ZnO nano rods, particle size was also detected by particle size analyzer. We found that when concentration of the precursor increased from (93%) to (97%) particle size of nano ZnO decreased from (80 nm) to (42 nm).

Keywords: ZnO nano rods, polymer derived ceramic.

Introduction:-

Polymer derived ceramics or (PDCs) is a technique used to produce ceramic from polymeric solutions, polymer to ceramic transformation is used for the production of nano oxide ceramic starting from a molecular precursor[1-4]. The polymeric molecular precursors are inorganicorgano-metallic systems that are designed to composed a ceramic material with a defined chemical composition[5], and a nano-scale organization by several heat treatment stages[6]. PDCs method produce a pure net shapes which are not known from the other ceramic production techniques, such as the production of binary systems of (Si₃N₄, and SiC,etc.)[7,8]. Ceramic materials produced by PDC technique exhibit enhanced thermomechanical properties and oxidation or phase separation up to (1600 °C)[9], due to the ultraregularity in their morphology[10]. From the other hand zinc oxide nanorods is a very important ceramic material, it is a semiconductor oxide with a (3.3 eV) band gap at room temperature, and a free excite binding energy of (60 eV) [11], these electronic properties beside its high thermo-mechanical properties make ZnO nanorods very widely used in several applications such as conductive oxide sensors, touch display equipment, beside optoelectronic devices such as solar cells panels[12]. ZnO nanorods are fabricated using chemical vapor deposition (CVD) or physical vapor deposition (PVD) from a metal-organic vapors[13], these techniques used a high growth temperatures up to (350-400 °C) and higher, while (PDC) technique can produce ZnO nanrods at temperature rates of (75-100 °C) depending on the type and concentration of the polymeric precursor. In this research we succeed to produce ZnO nano rods at room temperature (35°C) from an super ultra-saturation solution of zinc sulphate





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hydro acetate, we also investigate the effect of the precursor solution on morphology, diameter, and aspect ratio of the produced ZnO nano rods by XRD, and SEM.

Experimental part:-

Zinc hydro sulphate with (99.9 %) purity delivered from (MERCK, Germany), glacial acetic acid from (SAY Chemicals, India), and ultra-pure water from (Lomanio, China) were used as a starting material to form different concentrations of zinc sulphate hydro acetate.

To prepare ZnO nano rods , (20,22.2,24.4,26.6, and 28.8) gm of zinc hydro sulphate were dissolved in (25 ml) of glacial acetic acid to produce precursor solutions with (93 up to 97) concentration respectively, dissolving achieved using a magnetic stirrer type (DOLBY Ins, Netherland) at (200 rpm) for (5 hrs) at room temperature. The prepared super saturation solutions then were poured in a watch glasses, and they were left to dry over an ultrasound evacuated plate in an evacuated hood at (5 bar) pressure, and room temperature, hydrogen peroxide was used as a drying agent during the drying interval which was last for (12 hrs). A white layer were formed over the solutions it was isolated, washed with (5 %) methanol filtered and dried at (100 °C) to give a white powder which was test by XRD, and SEM to detect ZnO nanorods.

Results and Discussion:-

Polymer derived ceramic or (PDC) is a very special class of materials as they are synthesized via the polymer to ceramic transformation process of pre ceramic polymer precursors[3]. In the present work we succeed to produce zinc oxide (ZnO) nano rods with a hexagonal structure by the transformation of a super saturation solutions of zinc sulphate hydro acetate alkoxide precursor with (93,94,95,96,97) concentrations at room temperature, and a (5 bar) pressure under evacuated circumstances, hydrogen peroxide with concentration of (9%) was used as a reducing agent during the transformation process, after the transformation of the polymer precursors a white powder ere produced as shown in fig(1), after they been washed with methanol and dried in an autoclave they were tested with SEM, XRD, and particle size distribution analyser to detect the structure, mean diameter, direction of growth of the ZnO nano rods, and other properties.



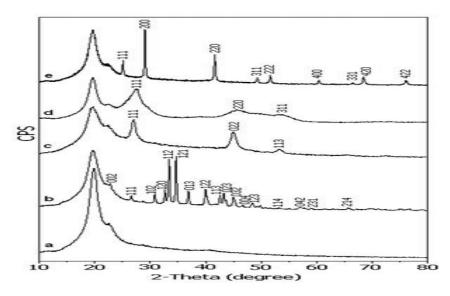
(Fig-1) The PDC ZnO powders from left to right (93,94,95,96,97)% precursor concentration

Figure (2) detect the XRD of the ZnO powders produced from the different concentration of zinc sulphate hydro acetate alkoxide precursor. First of all we may notice that all the patterns from (a to e) indicates the formation of zinc oxide ceramic ZnO which means that there is a total and clean transformation at room temperature for all the studied concentration of zinc sulphate hydro acetate alkoxide polymer precursor to an ultra-pure zinc oxide ceramic material and this mean alow cost and cold route that can be used for the production of ZnO. We also notice that peaks are largely depend on the concentration of the zinc sulphate hydro acetate alkoxide polymeric means that every concentration will form its unique structure and particle size.





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(Fig-2) XRD of the prepared ZnO nano rods (a)93%, (b)94%, (c)95%, (d)96%, and (e)97% precursor concentration

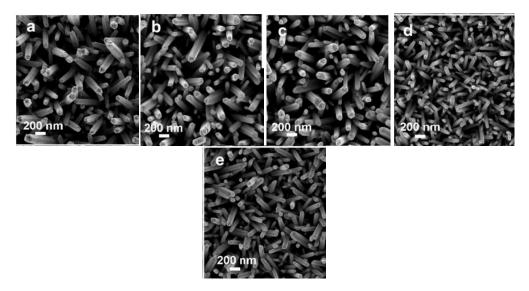
At the ordinary cases PDCs ceramic materials need high temperatures to complete the polymer precursor transformation, in this research we used the heat energy generated from the exothermic reaction between hydrogen peroxide (H₂O₂) to ensure the phase separation process of the free carbon and local formation of nano crystals as indicated in figure (2) which detect the XRD of ZnO powders produced from the different concentration of zinc sulphate hydro acetate alkoxide precursor. First of all we may notice that all the patterns from (a to e) indicates the formation of zinc oxide ceramic ZnO which means that there is a total and clean transformation at room temperature for all the studied concentration of zinc sulphate hydro acetate alkoxide polymer precursor to an ultra pure zinc oxide ceramic material and this mean alow cost and cold route that can be used for the production of ZnO. We also notice that peaks are largely depend on the concentration of the zinc sulphate hydro acetate alkoxide polymeric precursor which means that every concentration will form its unique structure and particle size. The splitting of the peaks that appear on the (b-e) patterns in XRD spectra is an indication that a material with a nano scale particle size has been formed, since splitting in peaks ensure that the material has transfer from its bulk state to the surface state, and a uniform growth of the ZnO ceramic oxide has been occurred as can be seen in Fig(3) which shows the SEM images for zinc oxide nano rods produced by PDC technique the direction of growth and hexagonal structure of the particles is very clear, we may also indicate that diameter of ZnO nano rods is largely affected by the concentration of the zinc sulphate hydro acetate alkoxide precursor the lower concentrations gives particles with high diameter this may be related to interval that the zinc sulphate hydro acetate alkoxide precursor needs to totally the of transferee to the finally ceramic material since the high precursor polymers need a lower time for the transformation and this mean a smaller ceramic particle size as can be seen from figure (3) that at (93,94, and 95)% precursor concentrations yield hexagonal ZnO nano rods, while (96,and 97)% yields the smallest ZnO nano rods particles. This nano domain structure of PDC ZnO oxide have been claimed to cause the super, combination of high other opportunity of properties such as optical, and electrical. The particle size distribution and mean particle diameter are illustrated in fig (4). Where the mean particle size decrease from (78nm) for (93, and 94) precursor concentration down to (62 nm) for (95, and 96) precursor concentration, and the lowest mean particle size is for the (97%) precursor which record a mean particle size of (42 nm).



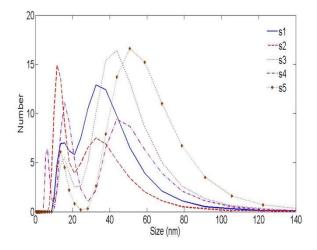


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ZnO nano rods that produce by polymer derived ceramic technique at room temperature provide a low cost nano ceramic oxide suitable for application including pharmaceutical such as (sun block) because of its high resistance to UV radiation, beside other several applications such as electronic liquid inks that are suitable for printing of electrical circuits and the batch production of low cost solar cells.



(Fig-3) SEM of the PDC ZnO nano rods (a)93%, (b)94%, (c)95%, (d)96%, and (e)97%



(Fig-4) Particle size distribution of PDC ZnO produced from precursor super saturation solution of zinc sulphate hydro acetate (S1) 93%, (S2) 94%, (S3) 95%, (S4) 96%, and (S5) 97%

4. Conclusions:-

During this research we can conclude that change of concentration of polymer precursor from (93 % up to 97%) lead to decrease ZnO nano rods particle size from (80 down to 42) nm at room temperature.

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