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Microwave Assisted Synthesis of $ZnCeO_2$ Nanoparticles and their Application

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Abstract:
Nanocrystalline $ZnCeO_2$ oxide synthesized by microwave assisted technique.The morphology of synthesized nanocrystalline $ZnCeO_2$ was characterized by scanning electronic microscopy (SEM) and X-ray diffraction. This is novel, rapid and simple method for synthesis of mixed oxides. The size of $ZnCeO_2$ found to be3nm. These synthesized photocatalyst applied for degradation of malachite green the photocatalytic degradation experiment was carried out by photoreactor under visible irradiation. The photocatalysts were studied with effect of time.
Sample were analysed by UV-visible spectrophotometer. The photodegradation efficiency of malachite green were observed high in presence of ZnCeO2. The degradation increases with time contact it is found 88% at 80 min.

Keywords: *Microwave Irradiation, Nanoparticals , XRD, SEM, Dye degradation*

1. Introduction

The mixed oxide of transition and inner transition metal (rare-earth) has a wide application of photocatalyst.To synthesis nanocrystalline different methods like sonochemically [1], [2], co-precipitation [3], solvothermal technique [4], sol-gel method [5], microwave synthesis [6] -[9] etc. Out of these microwave synthesis rapid more convienent.Microwave- heating is a transfer of electromagnetic energy to the thermal energy and is an energy conversion phenomenon rather than the heat transfer. The electric component of an electromagnetic field causes heating by two main mechanism dipolar polarization mechanism and conduction mechanism. In polarization mechanism for a substance to generate heat when irradiated with microwave is must possess a dipole moment in the water molecule. Dipole is sensitive to external electric field and will attempt to align itself with the field by rotation. The applied field provides energy for this rotation. As molecules vainly attempt to follow the field, they collide with one another and heating in the sample is observed high and low the frequency radiation does not give rise to efficient heating in the first case the field oscillates too quickly for the molecules to respond. In the second case the molecule follows the field so well that there is no random motion generated.

Recently a microwave assisted method has been widely applied in chemical reaction and synthesis of nanomaterials. Research has shown the method to be an attraction choice to promote reduction and is energy effective heating compared to convert heat conduction method due to the direct heating of the reaction mixture.By conventional method the mixture is heated and this then transfer the heat by convection.Microwave heating is more efficient in terms of the energy used produces higher temperature homogeneity and is considerably more rapid and conventional heat source. Mixed oxide is attractive for several reason such as high activity,good atomic efficiency and rapid response to changing condition in catalyst system [10]

In present study we employed the microwave method to synthesis of $ZnCeO_2$nanocrystalline mixed oxide.The properties of $ZnCeO_2$ nanoparticles were characterised by XRD diffraction and morphology of nanocrystalline were investigated by scanning electron microscopy(SEM). The photocatalyst applied for degradation of malachite green. The degradation of dyes were studied with parameter of time contact.

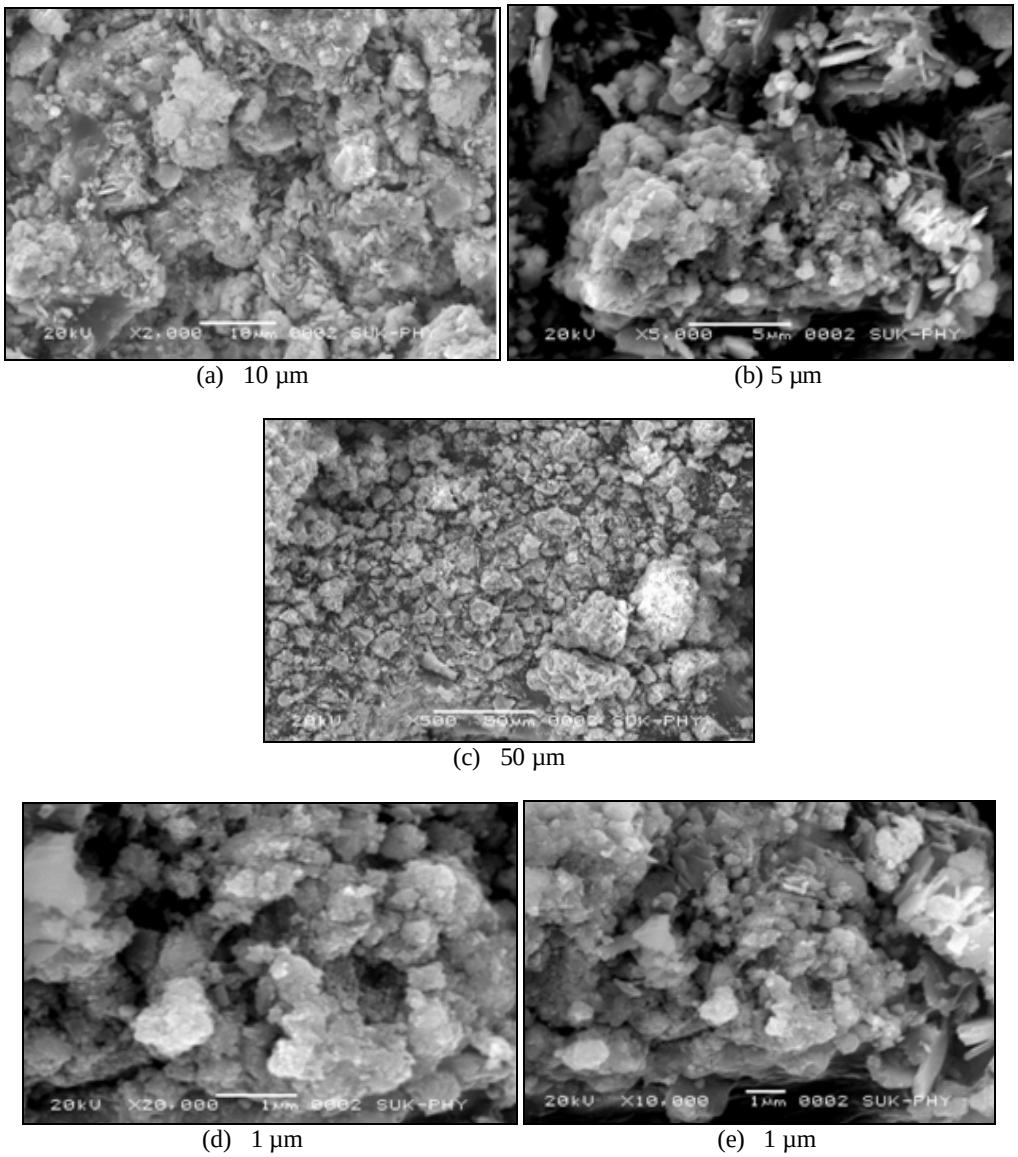
2. Experimental

All the chemicals used in the experiment were analytically grade. Ammonium ceric nitrate were purched from Lobachemie and zinc nitrate were purched from Thomas Baker company,India. $ZnCeO_2$ nanoparticles were synthesized by mixing zinc nitrate Zn $(No_3).6\ H_2O$ and ammonium ceric nitrate $(NH_4)_2NO_3$.Instoichiometic amount 1:1. These two chemicals were mixed throughly and grounded After grinding it transfered into a porcelain dish then these mixture was irradiated in microwave oven [Panasonic NN-GT221 W]for 2 minute.After irradiation observed pale yellow coloured crystals of $ZnCeO_2$ .Powder was washed with de-ionised water for several times to remove unreacted impurites.Then powder dried in oven at 70^oc about 3 hours. At the time of heating the evolution of large amount of gases with fumes occurs coming out from the exhaust opening provided on the top of the microwave oven.Synthesised powder characterized by SEM and XRD.

3. Result and Discussion

3.1. SEM image of the ZnCeO2

The $ZnCeO_2$ Nanoparticles are aggregated with small crystal.The scanning electron microscopy(SEM) revels that five types of crystal morphologies of $ZnCeO_2$ 10μm,5μm,10,1μ,50,um observed.The morphology of the synthesized $ZnCeO_2$nanoparticles is spherical and nano rod shaped are observed. It is shown in figures 1.



(a) 10 μm (b) 5 μm

(c) 50 μm

(d) 1 μm (e) 1 μm

Figure 1: SEM image of nanocrystlline $ZnCeO_2$

3.2. XRD Patterns of the Samples

The synthesized nanocrystalline $ZnCeO_2$ characterized by x-ray diffracation using curadiation(1.5406A0).The synthesized nanoparticles were pure and diffraction peak of any other impurities were not detected.All the diffraction peak are rather sharp which indicate the $ZnCeO_2$ sample has high degree of crystallinity.

The characteristic peak are observed at 2Θ values at 36.3 From XRD data crystalline size synthesized $ZnCeO_2$ particles was estimated by De-bye scherrers equation$Dp=0.9\lambda/ \beta\cos\theta$ whereDp is the size of particle in nm ,$\lambda$is wavelength of x-ray (1.5406) and β is the full width at half maximum (FWHM) and θ is the diffraction peak angle.XRD pattern of synthesied $ZnCeO_2$is shown in following fig.2

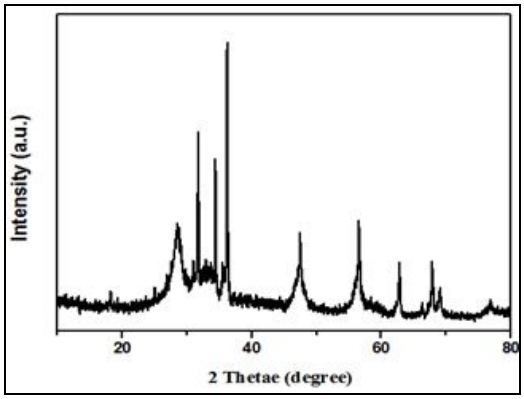


Figure 2: XRD pattern of synthesied $ZnCeO_2$nanocryastal

4. Application of Phtocatalyst

4.1. Photocatalytic activity of $ZnCeO_2$

The synthesized photocatalystZnCeO2 applied for degradation of malachite green .The photocatalytic degradation experiment was carried by photoreaction under visible irradiation and sample were analysed by UV- visible spectrophotometer. The photodegradation efficiency of malachite green estimated by following equation.

%degradation of days = $(1-A_t/A_0)x100$

$=(1-C_t/C_0)x100$

Where,$A_t$and A_0- theabsorbance of dye solution.

A_0-initial stage and

A_t- irradiation time

$C_0$and C_t- the concentration .

4.2. Effect of Time

Photocatalytic degradation experiments were studied at 10 ppm.Concentrationsolutionof dyes for different time intervals from 0min. to 80 min.The percentage ofdegradation increase with increase in time contact. 88% degradation of dyes was observed. Different time intervals and%degradation is represented in following table.

Sr .No	Time interval in min	Absorbance	% Dye degradation
1	0	1.63	0.0
2	20	1.025	37.12
3	40	0.468	71.29
4	60	0.265	83.75
5	80	0.124	87.55

Table 1: Effect of time on photocatalyticdegradiation rate

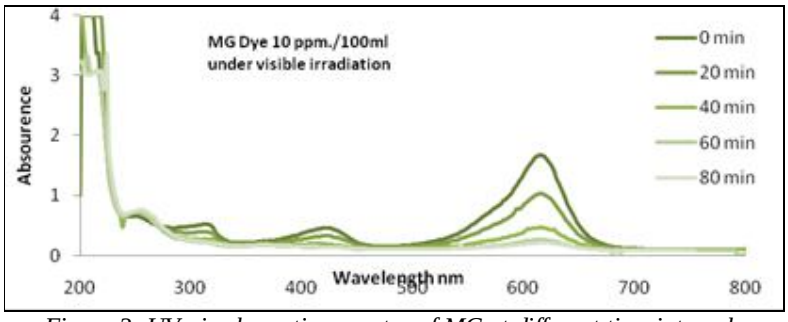


Figure 3: UV-vis absorption spectra of MG at different time intervals

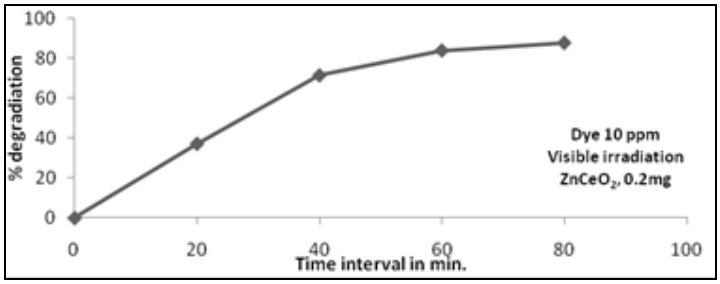


Figure 4: % degradation of MG at different time intervals

5. Conculsion

Microwave assisted method to synthesis $ZnCeO_2$has been successfully established this method is simple rapid and convenient and more efficient to control morphological and structural properties.The average partical size of nanocrystals calculated with XRD and it is found 3nm. It is expcted that the synthesized mixed oxide can find potential application in several field like photocatalyst.The rate of degradation was high at 80min.

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