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Exploration on Comprehensive Chemistry Teaching Experiment Converted by the Scientific Research Experiment

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ABSTRACT. This work investigated the design and conversion from scientific research experiment preparation and application of ZnO and Ce-ZnO micro-nano materials to a research-based comprehensive chemistry experiment for senior undergraduates. The ZnO micro-nano material in the hexagonal, plate like, and rod shapes was prepared by high-temperature calcination, direct precipitation, and hydrothermal synthesis, and analyzed their photodegradation of methyl orange. Through the study and optimization of all the steps of the experiment, the best experiment conditions, the most effective handling methods, and the appropriate reaction time were obtained. The experiment could satisfy the requirements of stability and reproducibility in teaching experiments. Through this comprehensive experiment, the students could consolidate the principle of ZnO micro-nano material, understand and grasp the working principle and operation skill of the vis spectrophotometer, muffle, reaction kettle, x-ray powder diffraction, and field-emission scanning electron microscopy, and develop their comprehensive ability including the related chemical basic knowledge, experimental operation, instrument analysis and relevant scientific literacy and research ability.

KEYWORDS: Comprehensive chemistry experiment, ZnO, Ce doping, Scientific experiments

1. Introduction

Chemistry experiment teaching occupies an important position in chemistry teaching, which is an essential way to deepen students' understanding of theoretical knowledge and improve their practical ability. Through the comprehensive chemical experiment practice, students are required to master the basic theory of chemistry and experimental operation ability, to break through the previous basic chemistry experiment for a single secondary discipline learning barrier. In this way, students can stand in the perspective of the first discipline of chemistry to think about problems, which is no longer simply repeat the operation on the book. Most universities combine new products, technologies, and materials to synthesize and translate the chemistry of target product innovations into experimental research from scientific experiments , chemical materials, equipment, experimental content, and teaching to maximize the promotion of students' comprehensive laboratory skills in chemistry, which can establish a new teaching mode of comprehensive chemistry experiments in a reasonable and effective way, cultivate the comprehensive experimental ability of college students, and improve the level of experimental teaching.

With the rapid development of industry and economy, global environmental pollution is becoming more and more serious [1]. ZnO is a multifunctional semiconductor material with a wide direct bandgap of 3.37ev and a large exciton binding energy of 60meV [2]. In addition, the doping of transition metal ions causes the bandgap to redshift and the absorption capacity of visible light is increasing [3, 4]. At present, many scholars in domestic and abroad have studied the ZnO micro-nanomaterial of different shapes such as nano-particles, nano-wires, nano-rods, and nano-films for different preparation technics [5] and explored the transformation of some scientific research experiments into comprehensive chemistry teaching experiments. Liao S. analyzed the effects of the molar ratio of Na_2CO_3 and $ZnSO_4 \cdot 7H_2O$, PEG-400 dosage, grinding time, pyrolysis temperature and pyrolysis time and other factors on the synthesis of ZnO crystal, and explored a new synthesis method for the target product ZnO, as well as an innovative comprehensive chemical experiment integrating chemical experiment and data mining technology [6]. Xu H. designed a comprehensive chemical experiment of *preparation of ZnO and photocatalytic degradation of methylene blue in water* with rich content, easy operation and strong practicability for students, and improved comprehensive chemical experimental ability through theoretical teaching and practical teaching [7]. The transformation of the above scientific research experiments into comprehensive experiments for students is still in the exploration stage, so a new comprehensive chemistry experiment library should be gradually established to meet the requirements of comprehensive chemistry experiments for undergraduates.

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In this paper, part of the achievements of the inorganic chemistry research experiment was transformed into research-oriented comprehensive chemistry teaching experiments. The target of an integrated chemistry teaching experiment is to produce ZnO micro-nano material by different experimental methods of high-temperature calcination, direct precipitation, or hydrothermal with basic operations such as washing, filtration, centrifugal separation, solution volume determination. During the experiment, students can fully understand the synthesis method and principle of ZnO micro-nano material, and master the operating methods of VIS Spectrophotometer, low-speed bench centrifuge, muffle furnace, reaction kettle, X-ray powder diffract meter, field-emission scanning electron microscopy, X-ray energy spectrum and other equipment. This comprehensive chemistry teaching experiment can cultivate the ability of independent thinking and innovation for students. The application prospect and development trend of ZnO micro-nano material in water pollution control can be understood through the literature review. The essence of scientific research experiments integrated into the teaching experiment is advanced and applicable.

2. Preparation of Zno Micro-Nano Material

In this experiment, ZnO-A, ZnO-B and ZnO-C nano materials are prepared by high-temperature calcination, direct precipitation and hydrothermal method, respectively. The synthesis process is shown in Figures 1 to 3. The optimal ZnO micro-nano material from ZnO-A, ZnO-B or ZnO-C by photo degradation performance of methyl orange is selected for Ce doping. In order to achieve the teaching requirements with stable phenomenon and good repeatability, the optimal experimental methods and reaction conditions for preparation of ZnO and Ce-ZnO micro-nano materials are explored.

(1) Preparation of ZnO-A (hexagonal prism) by high-temperature calcination

2.40g $Zn(NO_3)_2·6H_2O$ and 2.61g $C_4H_6N_2$ is dissolved in 96.4mL CH_3OH, respectively. The full dissolved $C_4H_6N_2·CH_3OH$ is rapidly poured into the $Zn(NO_3)_2·CH_3OH$, and strong stirring 60 min at room temperature. The precursor ZIF-8 is obtained that reaction liquid centrifuge for 10 min under 4000 r/min, abandon to supernatant fluid, will produce sediment with methanol solution of washing many times at 75℃ oven drying 10h. It takes to get ZnO-A that from ZIF-8 in muffle furnace calcin with 2h under a certain temperature.

(2) Preparation of ZnO-B (flake) by direct precipitation

7.22g ZnSO4·7H_2O and 4.80g NaOH is dissolved in 100ml distilled water, respectively. Then, 16ml PEG-400 is added to $Na_2SO_4·7H_2O$ solution, after being completely dissolved into NaOH solution while ultrasonic oscillations 2h at room temperature, and filtration after 20h. It takes to get ZnO-B that the sediment is respectively dissolved in C_2H_6O and distilled water washing three times to remove Na2SO4,PEG-400 and unreacted raw materials, and place in 80℃ drying oven for 4h.

(3) Preparation of ZnO-C (bar) by hydrothermal method

0.2190g $Zn(CH_3COO)_2·2H_2O$ and 0.1200g $C_6H_{12}N_4$ are placed in 100ml autoclaved bottle, and dissolved with water at the breaking point. The autoclave placed in 180℃ oven for 12h reaction, and centrifuged for separation. Deionized water is washed three times, and then placed in a vacuum drying oven at 40℃for 4h to obtain ZnO micro-nano material, denoted as ZnO-C.

3. Design of Comprehensive Chemistry Experiment

Course teaching of comprehensive chemistry experiment is a comprehensive and research-oriented major in our university for senior college students. This course aims to strengthen the comprehensive application for experimental methods in inorganic, organic, physical and chemical subjects, ability training of experimental skills, proficient modern analytical instruments and analytical mapping. In the process of experiment for students, cultivate the ability of innovative, independent design experiment and solve specific problems. In this paper, by exploring the preparation method of ZnO and Ce-ZnO micro-nano materials and their application research in inorganic chemistry scientific research experiment, the optimal reaction condition of reaction system and reasonable experimental operation time of the reaction system, the comprehensive chemistry teaching experiment with good design repeatability and meeting the experimental requirements of students was extracted.

3.1 Experimental Principle of Photocatalytic Degradation

The generation of electron-hole pair is due to the semiconductor catalyst by light irradiating, which produce the highly active photo-generated electrons e- with negative charge on the conduction band of semiconductor and photo-generated cavities h+ with positive charge on the valence band. Composite reactions and redox reactions occur when the electrons and holes reach the surface, recombination reactions and redox reactions occur. Hydrocarbons, halogenated organics, surfactants, dyes, pesticides, phenols, aromatic hydrocarbons and other organic pollutants are degraded by using highly active hydroxyl radicals. In this experiment, the catalyst is used semiconductor material of ZnO, and the reactant is methyl orange dye.

3.2 Chemicals and Apparatus

Chemical reagents and experimental apparatus involved in the preparation and photo catalytic degradation of ZnO and Ce-ZnO micro-nano materials are shown in Table 1.

Table 1 Chemical Reagents And Experimental Apparatus

Chemical reagents		Experimental apparatus	
Title	Symbol	Title	Symbol
2-Methylimidazole	$C_4H_6N_2$	Magnetic Stirrers	RH basic 2
Zinc acetate dihydrate	$Zn(CH_3COO)_2\cdot 2H_2O$	Centrifuge table low speed	TD25-WS
hexamethylenetetramine	$C_6H_{12}N_4$	Muffle furnace	SX2-4-10
methanol	CH_3OH	VIS Spectrophotometer	722S
ethanol	C_2H_6O	reaction kettle	YZHR-100
Methyl Orange	$C_{14}H_{14}N_3SO_3Na$	Ultraviolet high pressure mercury lamp	GGZ300
Rhodamine	$C_{21}H_{17}ClN_2O_3$	X-ray powder diffractometer(XRD)	D8 ADVANCE
methylene blue	$C_{16}H_{18}ClN_3S$	field-emission scanning electron microscopy(SEM)	S-4800
PEG-400	$HO(CH_2CH_2O)nH$	Energy Dispersive X-Ray Spectroscopy(EDX)	D8 ADVANCE

3.3 Experiment

In this experiment, ZnO and Ce-ZnO micro-nano materials are prepared and analyzed by high-temperature calcination, direct precipitation and hydrothermal method, respectively. It's to determine the photodegradation effect from time-degradation rate and dynamic curve by photodegradation experiment of methyl orange solution for different morphology ZnO micro-nano materials. The specific experimental steps are as follows:

(1) Preparation and characterization of ZnO and Ce-ZnO micro-nano materials

The synthesis process of ZnO and Ce-ZnO micro-nano materials is detailed in section 2. The microstructure and structural properties of ZnO micro-nano material is analyzed by experimental method of field-emission scanning electron microscopy (SEM), X-ray powder diffraction (XRD) and X-ray energy spectrum (EDX).

(2) Concentration–absorbance relationship

A 1000mg/L methyl orange solution is prepared and diluted 10 times to obtain a 100mg/L methyl orange solution. Methyl orange solutions of 1.00ml, 2.00ml, 3.00ml, 4.00ml and 5.00ml are taken out and dropped into 50mL volumetric bottle. The water volume is constant, and 2.00, 4.00, 6.00, 8.00, 10.00mg/L methyl orange solutions are obtained. The concentration-absorbance relationship is determined by absorbance of methyl orange solutions with different concentrations at the wavelength of 464nm.

(3) Measured of Absorbance for methyl orange by photodegradation

Two equal mass ZnO micro-nano materials are added to 50mL methyl orange solution with a concentration of 10.00mg/L, and the methyl orange reached adsorption/desorption equilibrium on the surface of the catalyst by magnetic stirring. One part of the reaction liquid is taken and continued to be stirred magnetically in the dark. The reaction liquid is removed 4mL/15min. After centrifugation, the supernatant is taken and analyzed by visible spectrophotometry. Another reaction liquid is put into the photodegradation unit with a light source of 300w high-pressure mercury lamp for photodegradation. The reaction liquid is removed 4mL/15min. After centrifugation, the absorbance of the supernatant at the wavelength of 464nm was measured. The degradation reaction ends.

(4) Time-degradation rate relationship

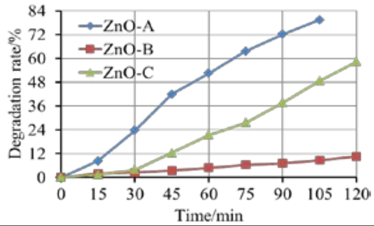
The concentration-absorbance curve and concentration difference of absorbance are obtained by above experiment. Then the degradation is calculated by absorbance-concentration change rate (concentration difference/initial concentration). The photodegradation curve for time-degradation rate is drawn.

3.4 Results and Discussion

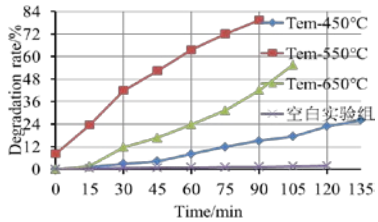
In order to explore the optimum reaction condition and the preparation method in the process of experiment. This experiment analyzes the photocatalytic performance of ZnO and Ce-ZnO micro-nano materials for methyl orange degradation by the influence of morphologies of ZnO with six prismatic, sheet and rod, and the calcination temperature of precursor ZIF-8 with 450℃, 550℃, and 650℃.

(1) Morphology

Figure 1(a) shows the time-degradation rate curve of methyl orange photodegradation by ZnO micro-nano material with different morphologies. It can be seen that the degradation rate of ZnO-A reached 72.11% after 90min of photodegradation. Relationship of degradation for ZnO-A, ZnO-B, and ZnO-C micro-nano material are ZnO-A>ZnO-C>ZnO-B. Micro-nano material of ZnO-A is the best photodegradation effect on methyl orange.



(a) Morphology



(b) Calcination temperature

Fig.1 Analysis of Influence Factor for Methyl Orange by Photodegradation

(2) Calcination temperature

Figure 1(b) shows the time-degradation rate curve of calcination temperature for photodegradation performance of methyl orange by ZnO-A micro-nano material. It can be seen that the optimal calcination temperature of ZIF-8 precursor is 550℃ for ZnO-A. Compared with the temperatures of 450℃ and 650℃, the degradation rate of the target product increases the fastest, and the final degradation rate reaches 79.44%.

4. Conclusions

This paper optimizes the design of inorganic chemistry scientific research experiment based on the scientific research experiment of *preparation and application of ZnO and Ce-ZnO micro-nano materials*. Experiment covers multiple knowledge and skills for principle of ZnO micro-nano material preparation, operation method of muffle furnace, principle and operation of VIS spectrophotometer, and data analysis. To train the comprehensive skills in basic chemistry experiments, ability to consult references, independently design and data analysis for students.The following conclusions are obtained:

(1) As the leader of the experiment, students independently completed the design of the experimental scheme and experimental steps, as well as the analysis of experimental results. The instructor, as the guide, mainly introduces the experimental background, principles and common methods and means to solve problems, so as to stimulate students' learning initiative and enthusiasm, and strengthen students' confidence in research and exploration.

(2) In the process of experiment, centrifuge and improper operation will lead to muffle furnace experiment equipment experiment data error and security problems. It is recommended to use the centrifuge during centrifugal separation, to balance on the balance precision centrifuge tube and reactants, tubes placed symmetrically in the rotating heads, until the weight on both sides meets the requirements of the equipment. When preparing ZnO micro-nano material by calcination method at high temperature, the muffle furnace experiment equipment used has a slow temperature rise. Preheating should be done before the experiment to prevent inadequate reaction due to insufficient temperature.

(3) The experimental steps of this experiment are complicated and time-consuming. It is suggested to divide this experiment into 4 people/groups to prepare three different shape ZnO micro-nano materials and conduct photocatalytic degradation experiments. After re-optimization, the experiment period can be compressed to 24 credit hours.

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