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# STUDYING THE OPTIMAL CONDITIONS FOR THE STABILITY OF ($ZNZRO_3$) PREPARED BY SOL-GEL METHOD

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**ABSTRACT**

In this research, the stability of ($ZnO$-$ZrO_3$) was studied using the sol-gel method in the presence of different stabilizers (Oxalic acid, Citric acid, Starch, CMC, pectin) the effect of the type of stabilizer, stabilizer amount, time of stabilizing, and temperature have been studied. thus, the optimal conditions for the formation and stability of the (ZnO-ZrO2) were determined. The best result was achieved using pectin as stabilizer in an amount (0.075 gr) and with a (72 h) stabilization time at 25C temperature.

**KEYWORDS:** $ZnZrO_3$, Sol-gel, Pectin, gel stability.

## 1. INTRODUCTION

PEROVSKITE oxides are one of the most widely investigated classes of materials due to their important physical properties in ferroelectricity, piezoelectricity, dielectricity, ferromagnetism, magnetoresistance, and multiferroics, which find a widely variety of applications in ferroelectric random access memories, multilayer ceramic capacitors, sensors and actuators, magnetic random access memories, and the potential new types of multiple-state memories and spintronic devices controlled by electric and magnetic fields.[1,2] Most perovskite oxides are still prepared by conventional solid-state reactions via the corresponding oxides or oxides and carbonates at temperatures over 1000°C. However, perovskite oxides prepared by the conventional solid-state reactions usually suffer from their particles with uncontrolled and irregular morphologies, which result in poor electrical properties of the sintered ceramics.[2] Recently, electroceramic materials have followed a similar trend to the miniaturization as the conventional semiconductor devices, the synthesis of nanosized oxidic building blocks now moves into the focus of scientific and technological interest.[3] In recent years, wet chemical methods have been developed to replace the conventional solid-state reactions for the synthesis of perovskite oxides, which can provide a molecular level mixing of the individual components, reduce the diffusion path in the nanometer range, and yield the final crystalline products at much lower temperatures.[4-6] Therefore, the size and morphology of the particles can be well controlled and metastable phases could be produced. As one of wet chemical methods, hydrothermal synthesis can provide an excellent approach to synthesize perovskite oxide nanoparticles under much milder conditions, which involves heating an aqueous suspension of insoluble salts in an autoclave at a moderate temperature and pressure so that the crystallization of a desired phase will take place. The advantages of hydrothermal crystallization are the reduced energy costs due to the moderate temperatures sufficient for the reaction, less pollution, simplicity in the process equipment, and the enhanced rate of the precipitation reaction.[7] Since there is no necessity for high-temperature calcination in this case, so the additional milling process is eliminated. Up to now, many perovskite oxide nanoparticles such as BaTiO3[8-13], PbTiO3[14-17], Pb(Zr,Ti)O3,18–21 (Ba,Sr) TiO3[22-24] have been synthesized by hydrothermal method. In comparison to these perovskite oxide nanoparticles, the preparation of perovskite nanosized ZnZrO3 powders has been much less investigated. Recently, it is reported that the nanosized ZnZrO3 powders synthesized by sol-gel method have intense photoluminescence UV emission, which has promising application (as photoelectrochemical working electrodes) in the dye-sensitized solar cells.[25-27] In addition, the effects of post-annealing duration and temperature on the structural and photonic properties of ZnZrO3 nanoparticles were also investigated. A significant red shift to visible region (from 394 to 413 nm) was observed as increasing the post-annealing duration and temperature. X-ray diffraction patterns also revealed that high post-annealing temperature could promote the formation of the ZnZrO3 nanoparticles, and a higher percent of ZnZrO3 was formed at 800°C (27.2% w/w) and maximum percent (37.7% w/w) was achieved at 1000°C for 120 min. However, the crystalline ZnZrO3 nanoparticles were mixed with other phases of oxides such as hexagonal wurtzite ZnO, cubic ZrO2, and monoclinic ZrO2, which were not useful for improving the catalytic activities of the synthesized products. To enhance the photoelectrochemical properties of ZnZrO3 powders, their phase purity, particle size and morphology should be well controlled. However, there are some difficulties to achieve the above goal via sol–gel process because a heat treatment at high-temperature over 600°C is required to remove the unreacted organics and to crystallize the ZnZrO3 powders in a sol–gel process. Therefore, we aim in this research to test a number of stabilizers and study the factors affecting the stabilization process with the aim of determining the optimal conditions for preparation of ($ZnZrO_3$) by sol-gel method.

## 2. EXPERIMENTAL

### 2.1. Materials And Apparatus

Zirconyl chloride $ZrOCl_2·8H2O$ (99% purity), Zinc nitrate $Zn(NO_3)_2.6H_2O$ (99% purity), ammonia, Oxalic acid, Citric acid, Starch, CMC, pectin, and distilled water. All chemicals used during the process of synthesis were purchased from Sigma-Aldrich, they were of analytical grade, and were used as received without any further purification.

### 2.2. Preparation of Zinc hydroxide

Zinc hydroxide was prepared by reacting ammonium hydroxide with a solution of zinc nitrate in a molar ratio (2:1) according to the following reaction:

$$Zn(NO_3)_2.6H_2O + 2NH_4OH \longrightarrow Zn(OH)_2 + 2NH_4NO_3 + 6H_2O$$

A white precipitate of zinc hydroxide formed.

### 2.3. Preparation of Zirconyl hydroxide

Zirconyl hydroxide was prepared by reacting ammonium hydroxide with a solution of Zirconyl chloride in a molar ratio (2:1) according to the following reaction:

$$ZrOCl_2.8H_2O + 2NH_4OH \longrightarrow ZrO(OH)_2 + 2NH_4Cl + 8H_2O$$

A white precipitate of Zirconyl hydroxide formed.

### 2.3. Formation of gel

The prepared hydroxides were mixed according to a molar ratio of (1:1) zinc hydroxide: Zirconyl hydroxide respectively, which is the ratio that leads to the formation of zinc zirconate ($ZnZrO_3$) based on the following chemical equation:

$$Zn(OH)_2 + ZrO(OH)_2 \longrightarrow ZnZrO_3 + 2H_2O$$

Different types of stabilizers were added each time to the mixture to studying the optimal stabilization conditions.

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of stabilizers type

Testing the effect of different types of stabilizers (Oxalic acid, Citric acid, Starch, CMC, pectin) by adding a fixed amount of them to the previously prepared mixture of hydroxides and measure the stability volume of the mixture as shown in the table.1.

**Table 1: Stability volume of gel using different stabilizers**

| Stabilizers | Oxalic acid | Citric acid | Starch | CMC | pectin |
|---|---|---|---|---|---|
| **Stability Volume (mL)** | 8 | 9 | 54 | 72 | 87 |

The results are represented according to the following graph

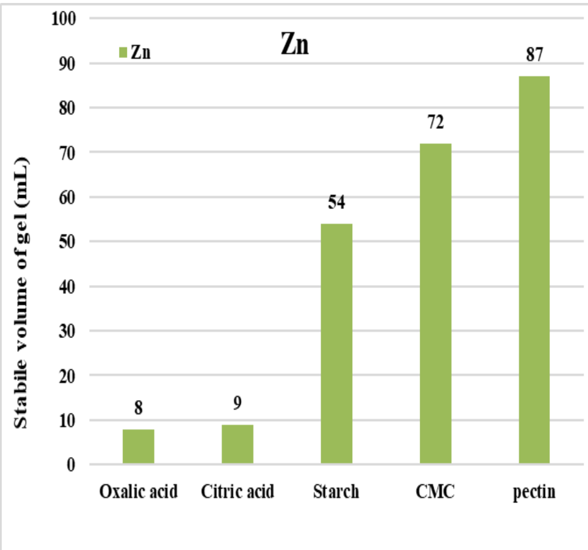


**Fig. 1: Stability Volume of Gel Using Different Stabilizers.**

It is noted that the highest stabilization volume was achieved using pectin stabilizer, Therefore, pectin is the best stabilizer for this mixture and will be used to study the remaining conditions.

### 3.2. Effect of pectin amount

To study the effect of the amount of pectin stabilizer, a group of previous hydroxides mixtures were prepared, and pectin stabilizer was added to each of them in increasing quantities. After that, the stability volume of (72h) was measured and the results were arranged in the table.2.

**Table 2: Stability volume of gel using different amount of Pectin.**

| Pectin amount (gr) | 0.005 | 0.01 | 0.02 | 0.05 | 0.075 |
|---|---|---|---|---|---|
| **stability volume (mL)** | 34 | 47 | 54 | 57 | 61 |

The relationship between the stability volume and the amount of pectin stabilizer was drawn in a graph shown in the figure.2.

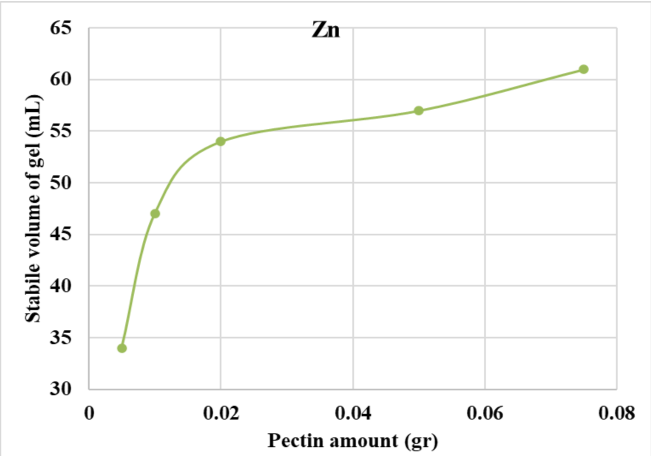


**Fig. 2: Stability volume of gel using different amount of Pectin.**

### 3.3. Effect of time on get stability

For studying the effect of time on gel stability, the stability volume has been measured for the mixture of hydroxides prepared in the presence of pectin as a stabilizer with (0.075 gr) for (96 h), the obtained results were arranged in the table.3.

**Table 3: Stability volume of gel at different times.**

| **Time (h)** | **1** | **2** | **4** | **8** | **12** | **24** | **48** | **72** | **96** |
|---|---|---|---|---|---|---|---|---|---|
| **stability volume (mL)** | 91 | 87 | 82 | 77 | 70 | 63 | 61 | 60 | 60 |

The relationship between the stability volume and the time was demonstrated in in the figure.3.

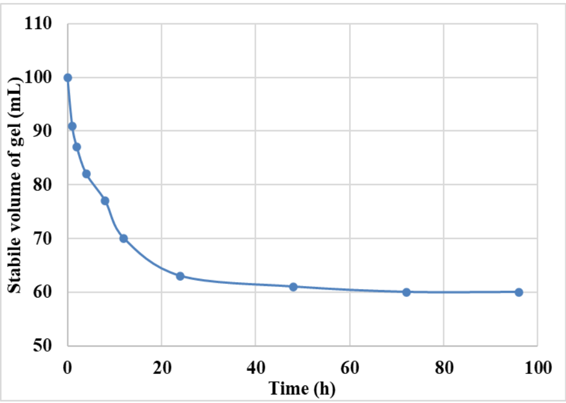


**Fig. 3: Stability volume of gel at different times.**

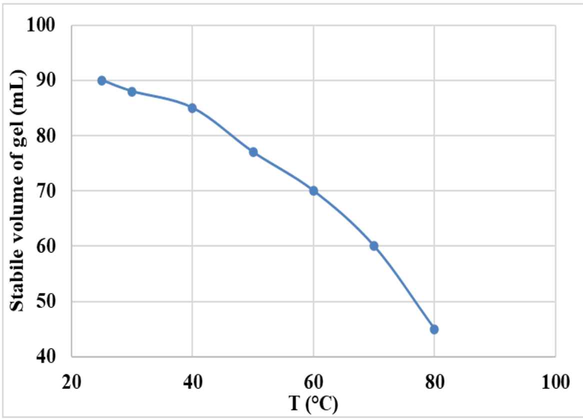


**Fig.4. stability volume of gel at different Temperatures.**

### 3.4. Effect of Temperature on get stability

For studying the effect of Temperature on gel stability, the stability volume has been measured for the mixture of hydroxides prepared in the presence of pectin as a stabilizer with (0.075 gr) at different temperatures after (1h) as shown in the table.4.

**Table 4: Stability volume of gel at different Temperatures.**

| **Temperature (°C)** | **25** | **30** | **40** | **50** | **60** | **70** | **80** |
|---|---|---|---|---|---|---|---|
| **stability volume (mL)** | 90 | 88 | 85 | 77 | 70 | 60 | 45 |

The relationship between the stability volume and the Temperature was demonstrated in in the figure.4.

## 4. CONCLUSION

The stability of (ZnO-$ZrO_3$) was studied using the sol-gel method in the presence of different stabilizers (Oxalic acid, Citric acid, Starch, CMC, pectin).

The effect of the type of stabilizer, stabilizer amount, time of stabilizing, and temperature have been studied. thus, the optimal conditions for the formation and stability of the (ZnO-ZrO2) were determined. The best result was achieved using pectin as stabilizer in an amount (0.075 gr) and with a (72 h) stabilization time at 25°C temperature.

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