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Solid Phase Synthesis of Methionine Complexes of Manganese Ion Assisted by Microwave

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Abstract: Methionine complexes of manganese ions were synthesized using the chelation technology under microwave irradiation. The influences of amount of water, reaction time, microwave power and the molar ration of methionine and manganese ions on the yield of the synthesized compounds were investigated. Results indicated that optimal synthesis conditions were amount of water, 10 wt %, irradiation time, 3 min, microwave power, 500 W, and the molar ration of reactant, 2:1, and the yield of the synthesized compounds was 82.1%.

Keywords: Microwave Irradiation; Solid State Reaction; Methionine Chelated Manganese

1. Introduction

Recently, the application of amino acid chelating metal ions has been reported. However, amino acid chelating metal ions is usually synthesized in solution, which has involved lots of disadvantages, such as longer synthesis time, higher cost, environment pollution, wastes of large quantities of organics solvent and complicated synthesis technology [1]. These become a bottleneck to use the chelation reaction. Compared with the traditional solution-state chemistry, solid-state synthesis via a route assisted by microwave irradiation is used to synthesis amino acid chelating metal ions. For example, Wang et al. reported that methionine complexes of iron ions were synthesized by employing a simple and novel microwave assisted solid state route in a microwave oven [2].

To the best of our knowledge, there have been no reports of microwave assisted solid state synthesis of methionine complexes of manganese ions in the literature. The purpose of this paper is to determine optimal synthesis parameters: amount of water, irradiation time, microwave power, and the molar ration of reactants to synthesis methionine complexes of manganese ions.

2. Experimental

2.1. Materials

Methionine and manganese sulphate were supplied by China medicine ShangHai reagent company; All other reagents were of analytical grade. The chemical reaction was carried out in a closed glass container containing 10 g reactants (methionine and manganese sulphate) and a little of water as initiator. The reaction mixture was then exposed to microwave irradiation in a domestic microwave oven for various time intervals with stirring. At the same time, airflow was introduced into a buffer bottle for timely removal of by-product sulfuric acid. After the reaction was completed, the products were finally dried in air to obtain the desired product.

Based on the single-factor experiment, the variables considered were amount of water, microwave heat time, microwave power, and the molar ration of methionine to manganese ions in the experimental design (Table 1).

Table 1. The levels of factors

Levels	Factors			
	Water A (wt %)	*Time B (min)*	*power C (W)*	*Amount of reactant D*
1	8	2	300	2.0:1
2	10	3	400	2.5:1
3	12	4	500	3.0:1

2.2. Measurements

Nitrogen content was determined according to the method of Kjeldahl described by Benton [3-4].

The synthesized compounds were analyzed for quantifying manganese ion content. Manganese ion concentration was determined by using the potassium permanganate titration method, then the chelation yield of the synthesized compounds was analyzed according to the formula:

Chelation yield % = the content of manganese in the synthesized compounds / total content of manganese × 100% (1).

3. Results and Discussion

In the experiment, water was used as an initiator. Fig.1 shows that the influence of the initiator on the yield of the synthesized compounds when other reaction conditions were as follows: irradiation time, 3 min, microwave power, 500 W, and the molar ration of reactant, 2:1. It could be demonstrated that the yield of the synthesized compounds increased with an increasing quantity of initiator. The yield of the synthesized compounds was close to the peak value when the quantity of initiator was set at 10 wt %. The percentage value started to decrease with the continuously increasing quantity of initiator. When the quantity of initiator increased, the concentration of reaction mixture decreased. Decreased concentration of reactants was disadvantage to the chemical reaction.

Fig.2 shows that the influence of microwave irradiation time on the yield of the synthesized compounds when other reaction conditions were as follows: amount of water, 10 wt %, microwave power, 500 W, and the molar ration of reactant, 2:1. Experimental results indicated that the yield of the synthesized compound gradually increased with increasing microwave irradiation time and reached approximately 80% when the irradiation time was 3 min. However, the trend of the yield change of the synthesized compound was small with increasing its irradiation time.

Fig.3 shows that the influence of microwave power on the yield of the synthesized compounds when other reaction conditions were as follows: amount of water, 10 wt %, irradiation time, 3 min, and the molar ration of reactant, 2:1. Experimental results indicated that the yield of the synthesized compounds gradually increased with increasing microwave power and reached approximately 82 % when the microwave power was at 500 W. After this point, the yield of the synthesized compounds started to decrease with increasing microwave power.

Fig.4 shows that the influence of the molar rations of reactant on the yield of the synthesized compounds when other reaction conditions were as follows: amount of water, 10 wt %, irradiation time, 3 min, and microwave power, 500 W. As shown in Fig.4, the yield of the synthesized compounds increased with the increase of the molar rations of reactant. The synthesized compounds achieved a maximum percentage of 82 % when the molar ration of reactant was 2:1. After this point, the yield of the synthesized compounds started to decrease with increasing their molar rations of reactant. If the molar ratio was too small, the complex formed was instable. In contrast, it would be a waste of complex amino acid, if the molar ratio was too large.

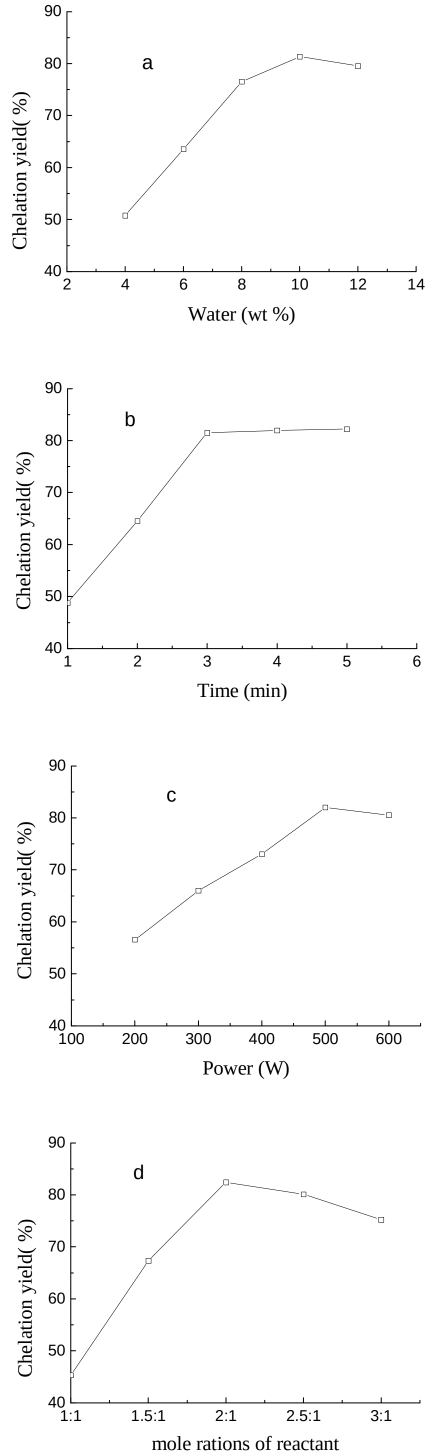

Figure 1. Variation of the Yield of the Synthesized Compounds With Amount of Water (a), Microwave Irradiation Time (b), Microwave Power (c), Molar Ration of Reactants (d) During the Reaction Process.

Table 2 shows that the results of the production of methionine chelated manganese ions according to the factorial design. In this experiment, the results revealed that the most relevant variable for the complexes production was amount of water and irradiation time. The effects of microwave power and the molar rations of reactant were not significant. In addition, percent of the synthesized compounds in No. 5 group reached maximum value 82.1%. Thus, the optimal conditions were approached using the model as follows: amount of water, 10 wt %, irradiation time, 3 min, microwave power, 500 W, and the molar ration of reactant, 2:1.

Table 2 The Data and Results of Experiments

Group	*A*	*B*	*C*	*D*	*Yield/ %*
1	1	1	1	1	78.80
2	1	2	2	2	82.00
3	1	3	3	3	81.60
4	2	1	2	3	80.90
5	2	2	3	1	82.10
6	2	3	1	2	81.00
7	3	1	3	2	79.20
8	3	2	1	3	81.70
9	3	3	2	1	82.40
K1	80.80	79.63	80.50	81.10	
K2	81.33	81.93	81.77	80.73	
K3	81.10	81.67	80.97	81.40	
R	3.02	4.01	2.02	0.90	

4. Conclusions

A high yield of methionine complexes of manganese was successfully achieved by employing a microwave assisted solid state route under the optimum reaction conditions, i.e., amount of water, 10 wt %, irradiation time, 3 min, microwave power, 500 W, and the molar ration of reactant, 2:1, and the yield of the synthesized compounds was 82.1%. It was illustrated that combination of microwave irradiation and solid state chemistry in the synthesis of amino acid complexes of manganese was very promising in the terms of mildness of reaction conditions, yields and time of reactions.

4. Acknowledgment

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