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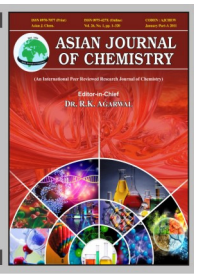

Study of Calcium Sulfate Whiskers

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The calcium sulfate dihydrate, magnesium chloride used for the crystal promotor, sodium lauryl sulfate used as dispersant in study of the hydrothermal synthesis process of calcium sulfate whisker factors. The concentratino of slurry was examined and fueling dispersant on the reaction. Analysis of morphology of calcium sulfate whisker and composition of the line by scanning electron microscopy and whisker growth mechanism was discussed.

Keywords: Hydrothermal synthesis, Aspect ratio, Crystal promotor.

INTRODUCTION

Whisker grows out of a short cut fiber in the form of monocrystalline under the condition of manual control. It is characterized by the uniform cross-section, the complete appearance and highly perfect inner structure. Compared with the glass fibre, most obvious advantages of the whisker lie in its high strength, small size, its fine appearance and even the whisker is easy to conbine with resin, rubber, plastic and other organic polymer compounds[1-4]. Calcium sulfate whiskers with raw material of gypsum which rich plaster in China mineral as sources are successfully developed. It is the lowest production cost, the greatest market demand, a strong market competitiveness and makes large numbers of applications possible among all whiskers[5].

The growth of whisker is under the unforced condition of water and heat. The nucleation, growth and growth rate of each crystal surface family are influenced by the reaction temperature, heating rate, pressure, medium, numbers and kinds of additives. It is obvious to present on changes of morphology, the formation and growth of calcium sulfate whiskers comply with the common law of growing[6-8].

The study mainly achieves the goal of controling the shape by changing its physicochemical conditions and examines the effects of the reaction temperature, the slurry concentration, the Ca/Mg ratio and numbers of dispersant added on the whisker morphology and aspect ratio.

EXPERIMENTAL

The experiment takes the calcium sulfate dihydrate as raw material and add optimum concentration of the solution of dilute sulfuric acid to make the solvent of certain mass fraction. Then adding anhydrous magnesium chloride for crystal promotor and sodium lauryl sulfate as dispersant and stirring them. Finally, the mixure under the hydrothermal reaction is placed in the autoclave after intensive mixing. Calcium sulfate whiskers are prepared by cooling, filtering, washing, extraction filtration and ariding after a period of time[9,10].

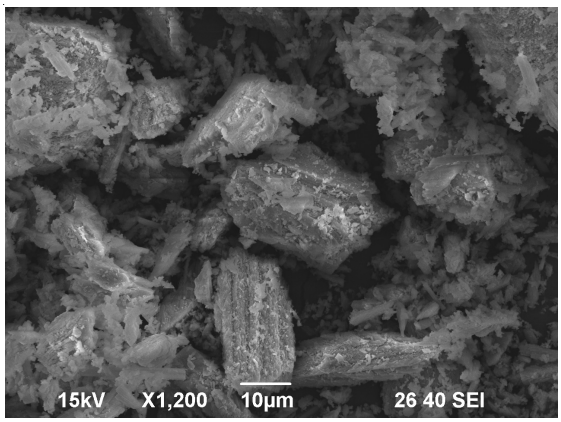
Observation of crystal form and measurement of length and diameter by the scanning electron microscopy and electron microscope to achieve the draw ratio of calcium sulfate whiskers.

Methods of analysis: Observation of crystal form and measurement of length and diameter by the scanning electron microscopy and electron microscope to achieve the draw ratio of calcium sulfate whiskers. Analysis of the product components by the thermogravimetry. Titration method for the determination of calcium content.

EMS diagram of materials: Take a small amount of raw materials were tested by EMS and achieve the pictures of morphology and structure in Fig. 1.

The EMS diagram of materials and the crystal morphology of raw materials has no certain rules. The end face structure almost can not be seen, and thus, the appearance is untidy.

Changes of slurry concentration: The whisker morphology, structure and conversion rate of raw materials were also influenced by the slurry concentration. Different kinds of weight of gypsum powder were measured and water was added to make 1, 2, 3, 5 suspension to control the Ca/Mg ratio as 12 and the reaction temperature as 120 °C. The EMS diagrams were shown as Figs. 2-5.



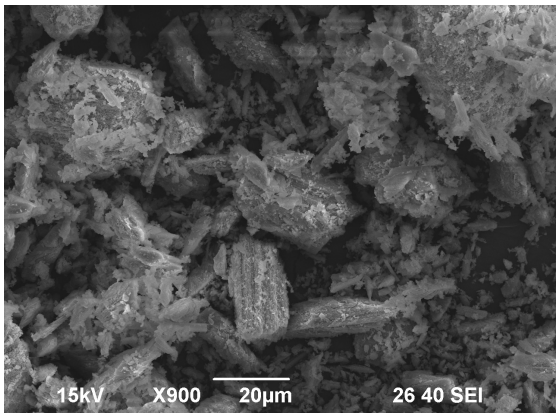


Fig. 1. EMS diagram of materials

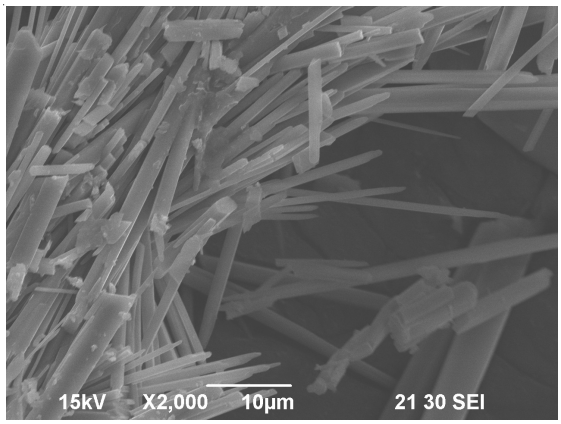


Fig. 2. EMS diagram of the concentration of 1%

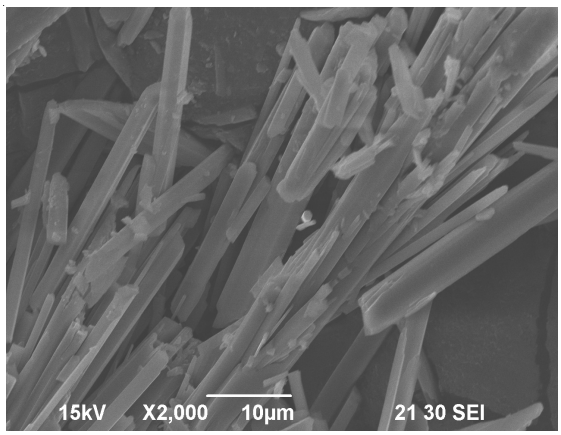


Fig. 3. EMS diagram of the concentration of 2 %

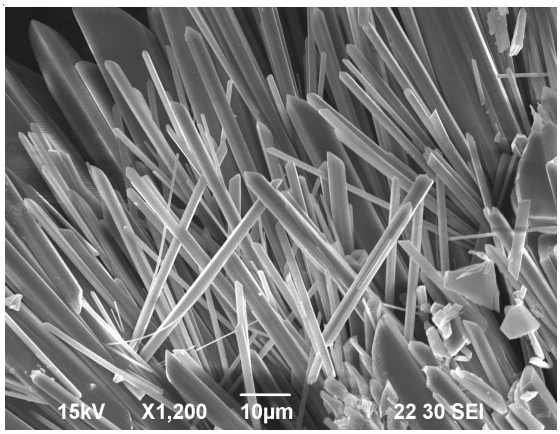


Fig. 4. EMS diagram of the concentration of 3 %

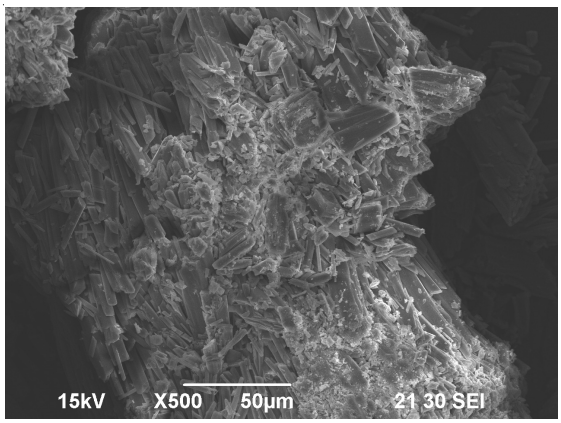


Fig. 5. EMS diagram of the concentration of 5 %

Different Ca/Mg in the reaction: Two grams gypsum was weighed in the experiment, 64.67 mL were added and the concentration of 3 % suspension was made to control the reaction temperature at 120 °C and **Ca/Mg** ratio as 10, 11, 12 and 13. The EMS diagrams of products achieved are shown in Figs. 6-9.

Effects of dispersant: Two grams gypsum was weighed in the experiment, 64.67mL were added and the concentration of 3 % suspension was made to control the reaction temperature at 120 °C and **Ca/Mg** ratio as 12. The sodium lauryl sulfate of different concentration which were 0.027, 0.077, 0.093, 0.120 % respectively was added and the EMS diagrams of products are shown in Figs. 10-13.

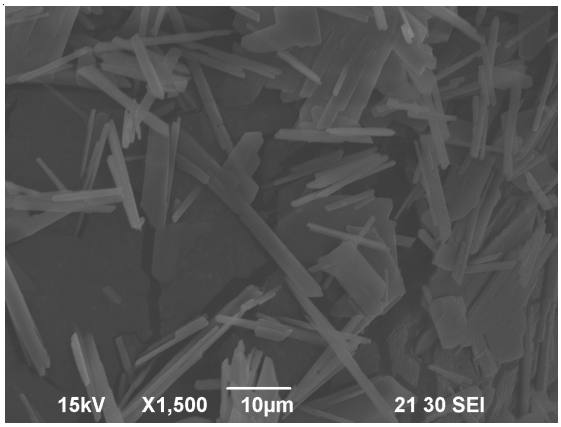


Fig. 6. EMS diagram of **Ca/Mg** of 10

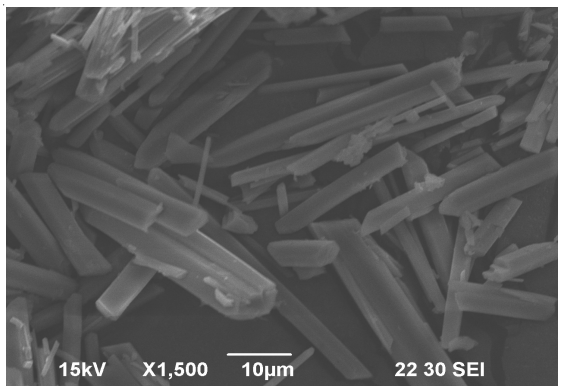


Fig.7. EMS diagram of **Ca/Mg** of 11

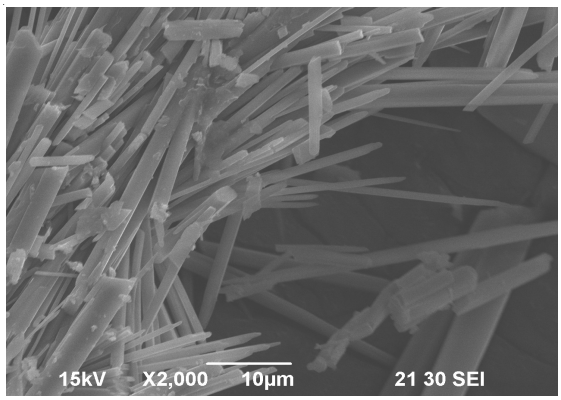


Fig. 8. EMS diagram of **Ca/Mg** of 12

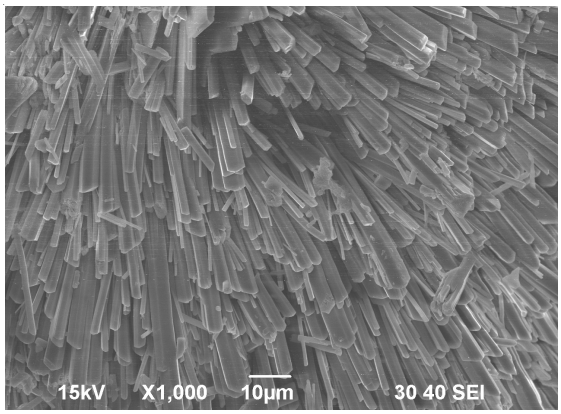


Fig. 9. EMS diagram of **Ca/Mg** of 13

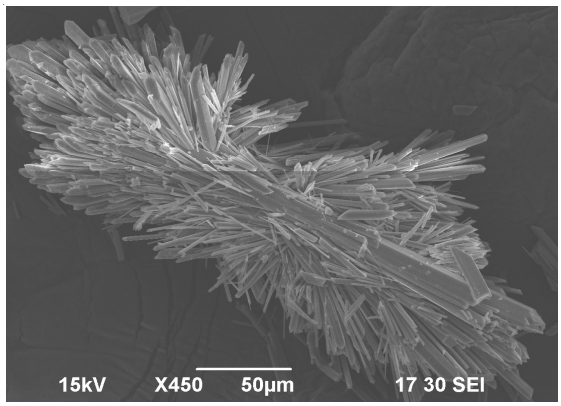


Fig. 10. EMS diagram of dispersant of concentration of 0.027 %

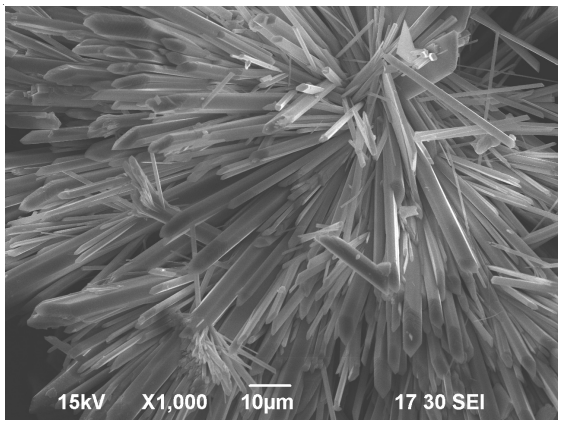


Fig. 11. EMS diagram of dispersant of concentration of 0.077 %

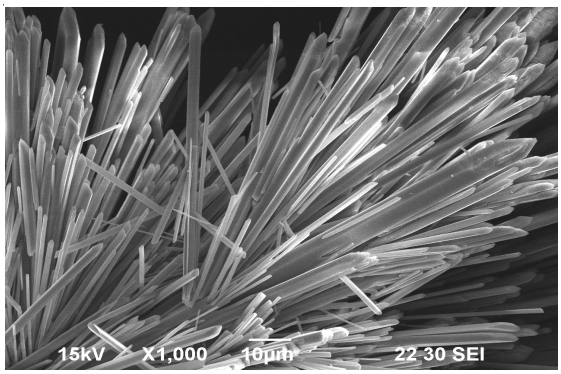


Fig. 12. EMS diagram of dispersant of concentration of 0.093 %

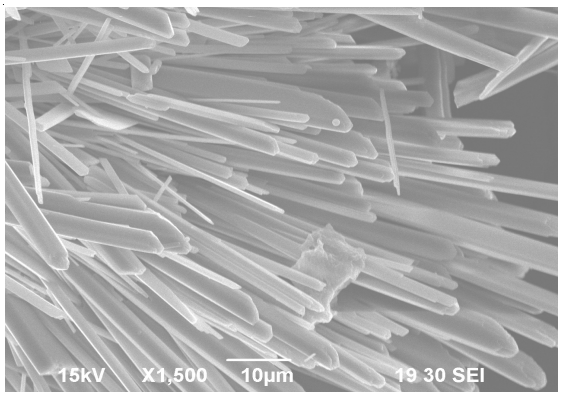


Fig. 13. EMS diagram of dispersant of concentration of 0.120 %

Comprehensive experiments: When the reaction temperature was 120 °C, the reaction time was about 1 h, the concentration of gypsum materials was 3 %, the Ca/Mg mole ratio was 12 and the concentration of sodium lauryl sulfate was 0.08 %. The EMS diagram of products achieved is shown in Fig. 14.

RESULTS AND DISCUSSION

Effects of the concentration of the raw material solution

(1) The formation of calcium sulfate whiskers has two processes of nucleation and growth. There were two kinds of growing speed: the aggregation velocity and directional velocity. The aggregation velocity is fast while the directional

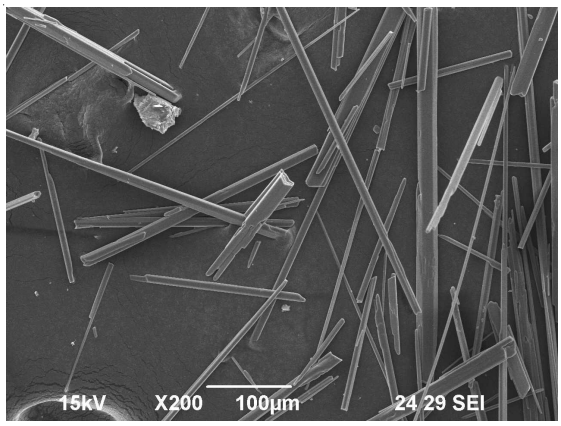


Fig. 14. EMS diagram of comprehensive experimental products

velocity is slow. The precipitation was aggregated by ions slowly, the lattice arrangement with adequate time and the crystalline precipitation was produced. The aggregation velocity V and supersaturation level of the solution followed the empirical formula:

$$V = k(Q-S)/S$$

where, V represents the initial velocity of the precipitation or the aggregation velocity; Q represents the concentration of the precipitation produced the moment the precipitant was added; S represents the solubility of the precipitation; Q-S represents supersaturation level of the precipitation; (Q-S)/S represents relative supersaturation level; K represents the constant of proportionality.

From the above formula, the aggregation velocity was fast initially then slow and the possibility of forming the crystalline precipitation with the relative supersaturation level of the solution more and more big.

(2) The extent of the concentration and the slurry concentration, whiskers grew with no adequate growing space led to the decline of aspect ratio. In addition, excessive crystal nucleuses existed in the system and insoluble calcium sulfate powders also deposited on the crystal nucleus to lead to the final product as a mixture of few short cut whiskers and much calcium sulfate.

From EMS diagrams of the products, it is clear that the overtop slurry concentration led to the decline of the feedstock conversion rather than the decline of the aspect ratio. The quality of whiskers was still excellent, but it had effects on the consumption of products. According to the solubility of calcium sulfate, the optimal slurry concentration was about 3 %.

Effects of crystal promotor on whiskers: EMS diagrams of the products showed that the magnesium chloride for the crystal promotor can make whiskers grow unidirectionally, changes of Ca/Mg ratio also affected the morphology of whiskers. The aspect ratio is biggest when Ca/Mg mole ratio was about 12.

Effects of dispersant on whiskers: EMS diagrams of the products suggest that sodium lauryl sulfate as dispersant can make whiskers caked disperse and the adding numbers also affected the morphology of whiskers. The caking of products was so serious when no dispersants added, which may affected the performance of products. The aspect ratio of whiskers were much bigger with the increase of numbers of dispersants in the extent, but the ratio needed to control otherwise it would affected the purity of products. The results analysis of the products showed that the concentration of dispersants was fine between 0.05 and 0.10 %.

Conclusion

Raw material of calcium sulfate dihydrate, magnesium chloride for the crystal promotor, calcium sulfate whiskers were prepared by hydrothermal synthesis process, the morphology of whiskers could be controlled. From the results of the products, when the reaction temperature was 120 °C, the reaction time was about 1 h, the slurry concentration was 3 %, the Ca/Mg mole ratio was 12 and the concentration of sodium lauryl sulfate as dispersant was between 0.05 and 0.10 %, the conversion ratio of calcium sulfate was high and aspect ratio was from 50 to 100.

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