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Original Research Paper

Effect of ethylenediaminetetraacetic acid (di sodium salt) and aquasoft 330 on crystal growth and morphology of calcium oxalate

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ABSTRACT

Calcium oxalate was synthesized from calcium chloride solution using oxalic acid in the presence of di sodium salt of ethylenediaminetetraacetic acid (EDTA) and commercially available1-hydroxyethylidene-1,1-diphosphonic acid (HEDP), aquasoft having chelating value 330 (AQ 330). The experiments were carried out at three different temperatures 60, 80 and 100 °C. The samples were characterized using scanning electron microscope (SEM), powder X-ray diffraction analysis (XRD) and Fourier transform infrared spectroscopy (FTIR) techniques. The morphological studies reveal that the tendency for agglomeration increases with increase in temperature. Interpretation of XRD pattern confirmed that whewellite with monoclinic structured is the most favored structure in the presence of EDTA where calcium oxalate hydrate having orthorhombic structure is most favored structure in the presence of AQ 330.

1. Introduction

Oxalic acid is an organic compound having chelating and reducing properties. Oxalic acid's main applications include cleaning or bleaching, especially for the removal of rust waste water treatment and as reducing agent. Another important usage of oxalic acid is as a mordant in dyeing processes. It forms a coordination complex with the dye which then attaches to the fabric or tissue.

1-Hydroxy ethylidene-1,1-diphosphonic acid (HEDP) is an organophosphoric acid corrosion inhibitor. HEDP shows excellent scale and corrosion inhibition effects below 250 °C. HEDP is chemically stable under high pH value, difficult to be hydrolyzed, and never decompose under ordinary conditions. HEDP can react with metal ions in water system to form hexa-element chelating complex, particularly with calcium ion. When used together with other water treatment chemicals, it shows good synergistic effects. HEDP is used as scale and corrosion inhibition in circulating cool water system, oil field and low-pressure boilers in fields such as electric power, chemical industry, metallurgy, fertilizer, etc. In dyeing industry, HEDP is used as peroxide stabilizer and dye-fixing agent.

Calcium oxalate has received considerable attention due to its presence in kidney stones. It is considered as the major constituent and cause for the formation of kidney stones. Three forms of calcium oxalate; calcium oxalate monohydrate (COM), calcium oxalate dihydrate (COD), and calcium oxalate trihydrate (COT) are known to exist. The most thermodynamically stable form of calcium oxalate is its monohydrate and hence it is the most common form found in the inorganic matrix of a kidney stone [1].

The acid-rich urinary proteins depress the crystallization of calcium oxalate even under supersaturated conditions [2] thus preventing the formation of stones. Many researchers have reported that preferential crystallization of different forms of calcium oxalate can be accomplished by usage of certain synthetic and natural molecules, such as polypeptides [3,4], sodium diisooctyl sulfosuccinate [5], poly(ethyleneglycol)-block-poly(methacrylic acid) [6], renal epithelial cells [7,8], poly(sodium 4-styrene-sulfonate) [9], bio polymeric additives [10], and protein isolated from bean seed coats [11]. Kato et al. have reported that the conventional homogeneous precipitation results into mono hydrate and granular calcium oxalate where as hydrolysis of oxamic acid catalyzed by enzyme (hydrolase) generate trihydrate and fibrous calcium oxalate [12].

Studies on the morpho-synthesis of $CaCO_3$ reveal that crystalline structure of calcium carbonate particles mainly depend on the precipitation condition, such as pH, temperature and presence of sequestrants [13,14] etc. Many studies have shown that a wide range of additives can influence the shape of crystalline $CaCO_3$ [15–18]. However the industrial applications of chelating agents such as EDTA, HEDP along with oxalic acid particularly in dyeing industry and the significance of calcium present in the water has not received required attention. The effect of the presence of calcium ions in water on the formation of calcium oxalate while using oxalic acid has not been reported so far.

This paper describes the significance of two chelating agents EDTA and AQ 330 on the morphology and structure of calcium

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