Materials Chemistry and Physics 114 (2009) 14–17

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Materials science communication

Habit and morphology study on the palm-based 9,10-dihydroxystearic acid (DHSA) crystals

Gregory F.L. Koay[a,b], Chuah Teong Guan[a,c,*], Sumaiya Zainal-Abidin[d], Salmiah Ahmad[b], Thomas S.Y. Choong[a]

[a] Department of Chemical and Environmental Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia
[b] Advanced Oleochemical Technology Division, Malaysian Palm Oil Board (MPOB), 43650 Bandar Baru Bangi, Selangor Darul Ehsan, Malaysia
[c] Institute of Tropical Forest and Forestry Products (INTROP), Universiti Putra Malaysia, 43400 UPM Serdang, Selangor Darul Ehsan, Malaysia
[d] Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, 25000 Kuantan, Pahang Darul Makmur, Malaysia

ARTICLE INFO

Article history:
Received 30 June 2008
Received in revised form
15 September 2008
Accepted 23 September 2008

Keywords:
Crystal structure
Crystallization
Electron microscopy
X-ray diffraction topography

ABSTRACT

Dihydroxystearic acid (DHSA) and its derivatives are hydroxyl fatty acids suitable to be used as multipurpose intermediates in the synthesis of personal care products and decorative cosmetics. In Malaysia, DHSA has been successfully produced from palm-based oleic acid, via epoxidation with per-formic acid followed with hydrolysis of the epoxide. The objective of this paper is to study the crystals of DHSA produced under different crystallization conditions. The crystal habit and morphology were observed in terms of scanning electron microscopy (SEM) and X-ray diffraction (XRD). Results show that solvent type, solvent concentration and cooling mode affect the crystal habit but not the morphology. The DHSA crystals agglomerated into either sphere-like or plate-like habit structure while always maintaining the triclinic crystal system.

1. Introduction

Hydroxyl fatty acids are highly sought after raw materials, especially in the personal care and cosmetic industry, due to their different behavior as compared to ordinary fatty acids [1]. However, due to the absence of castor oil, the main source of hydroxyl fatty acids supplied by nature, Malaysia has come out with dihydroxystearic acid (DHSA) derived from a low value byproduct of the palm oil industry known as oleic acid [2,3] as a compatible, suitable and economically feasible alternative.

DHSA, a type of hydroxyl fatty acids, was produced from epoxidation of oleic acid with either per-acetic or per-formic acid. The epoxide was then hydrolyzed in an aqueous solution resulting in 9,10-DHSA. The presence of hydroxyl and carboxyl groups in DHSA provided various reaction sites for the preparation of many useful derivatives. Studies have shown that DHSA is suitable as thickener, gelling agent, binding agent, mechanical properties booster and pigment dispersion enhancer in the formulation of decorative cosmetics [4–7].

In this work, solvent crystallization was carried out on crude DHSA so as to produce purified DHSA, conforming to the stringent non-irritant requirement by the cosmetic industry [3,8,9]. There is no literature report on the crystal study of DHSA. Thus, the objective of this paper is to illustrate the habit and morphology properties of the crystals produced when crude DHSA is solvent crystallized to remove the imbedded impurities, such as octanoic and decanoic acids.

2. Materials and methods

2.1. Materials

The preparation of crude DHSA was carried out according to the procedure patented by Awang et al. [2] with some minor modifications. The epoxidation process was carried out using per-formic acid rather than per-acetic acid. Palm-based oleic acid of 70% purity was purchased from Acid Chem Pvt. Ltd. Other chemicals used were: technical grade hydrogen peroxide of 50% purity; technical grade per-formic acid of 94% purity; technical grade sulfuric acid of >51% purity. These chemicals were used without any further purification and/or treatment. The crude and purified DHSA specifications are shown in Table 1. For solvents, technical grade ethyl alcohol and isopropyl alcohol were obtained from Chemi Industries (Malaysia) Pvt. Ltd. Dilution of solvents was carried out using reverse osmosis water. Nylon fabric filter cloth of 25 μm pore size was provided by Jaya Filter Pvt. Ltd.

2.2. Preparation of purified DHSA crystals

Crude DHSA was melted in oven preset at 80 °C for 8 h to ensure the total absence of small crystalline which could act as seeds. The molted crude DHSA was then mixed

* Corresponding author at: Department of Chemical and Environmental Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400, Serdang, Selangor, Malaysia. Tel.: +60 3 89466288; fax: +60 3 86567120.
E-mail addresses: chuah@eng.upm.edu.my, tgc615@hotmail.com (C.T. Guan).

0254-0584/$ – see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.matchemphys.2008.09.069

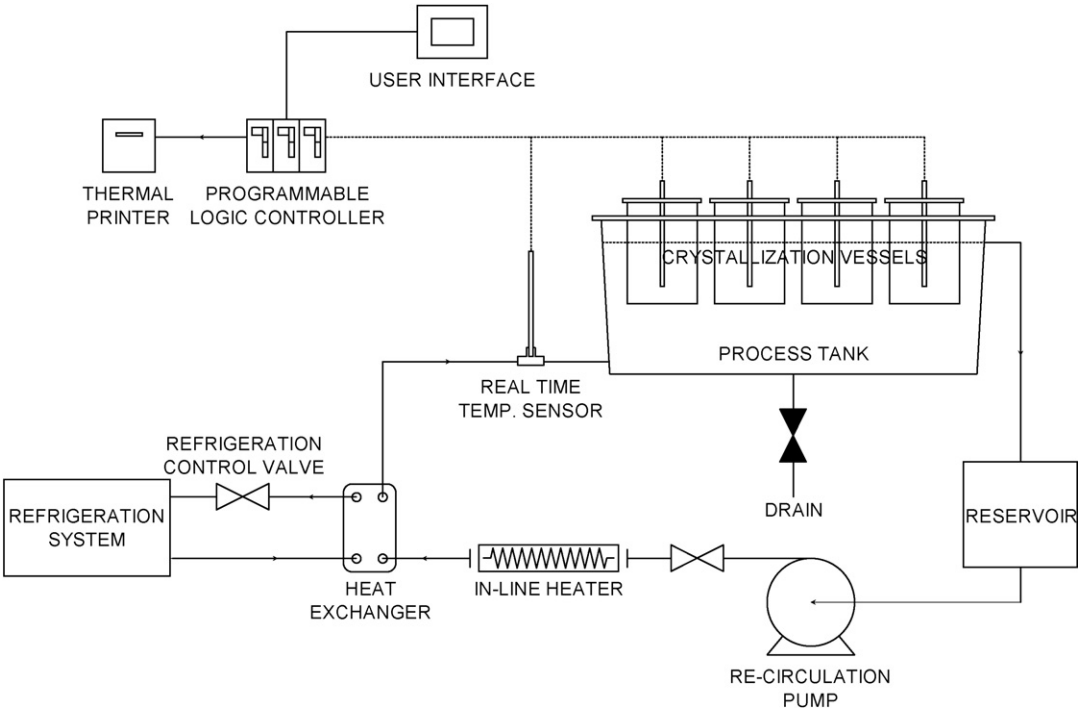

Fig. 1. Schematic diagram of the custom-fabricated simultaneous batch crystallization unit.

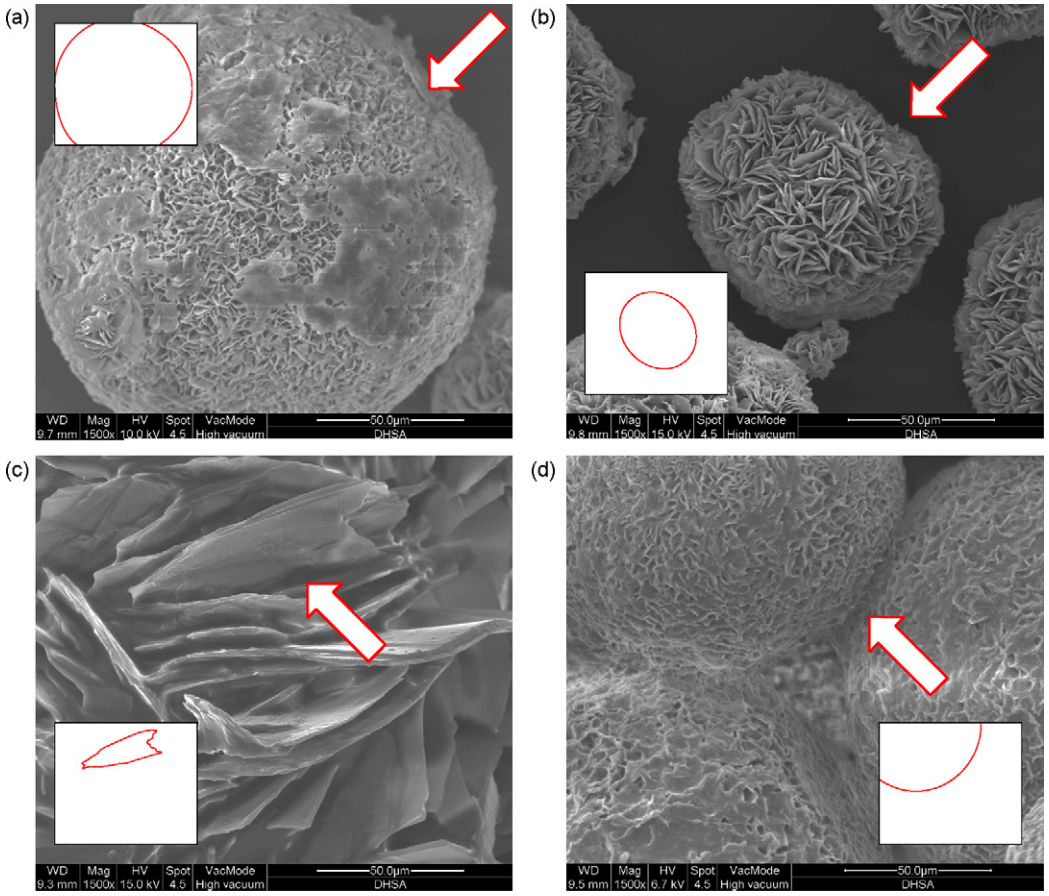

Fig. 2. SEM images of DHSA crystals obtained with ethyl alcohol: (a) 100% mass concentration, slow cooling, large spherical structure; (b) 100% mass concentration, rapid cooling, small spherical structure; (c) 90% mass concentration, slow cooling, large plate-like (flaky) structure; (d) 90% mass concentration, rapid cooling, large and dense packed spherical structure.