Dyes and Pigments 105 (2014) 137–144

Contents lists available at ScienceDirect

Dyes and Pigments

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

Crystallization and morphology of indanthrone converted from latent pigment in the solution with photo acid generator

Tang-Hao Liu [a], Wen-Tung Cheng [a,*], Kou-Tung Huang [b]

[a] Department of Chemical Engineering, National Chung Hsing University, Taichung 40227, Taiwan
[b] Material and Chemical Research Laboratory, Industrial Technology of Research Institute, Hsinchu 310, Taiwan

ARTICLE INFO

Article history:
Received 11 October 2013
Received in revised form
13 January 2014
Accepted 24 January 2014
Available online 7 February 2014

Keywords:
Latent pigment
Indanthrone
Morphology
Crystal structure
Photo acid generator
Acidolysis

ABSTRACT

The crystal phase, particle size, and morphology of organic pigments play an important role on the surface coating properties, such as substrate absorbency, surface smoothness, the light scattering properties, gloss, roughness, coating power, rheology and stability upon storage, etc. This paper aims to study the effect of photo acid generator (PAG) containing the stibium ion on the crystal structure and morphology of the regenerated pigment from the precursor of indanthrone in organic solvent. As measured from ultraviolet–visible spectroscopy and Fourier transform infrared spectroscopy, it was discovered that adding PAG accelerated the conversion of latent pigment to regenerated pigment. In addition, field emission scanning electron microscopy, energy dispersive spectroscopy and X-ray diffraction analysis indicated that, as the amount of PAG increased, the regenerated pigment lost its crystal structure, which particle size is increased from 1 to 4 μm and morphology is changed from a bar like form to a cubic and spherical form.

1. Introduction

In many applications, pigments were blended with polymeric matrix, which affect the surface coating properties, such as substrate absorbency, surface smoothness, the light scattering properties, gloss, coating power, rheology and stability upon storage, etc. [1]. Organic pigments are insoluble in the application medium, so the dispersion played an essential role in significantly influencing the quality and properties of the products in coating materials. Conventionally, treating the pigment through grinding, milling, or using a solvent was necessary to improve the dispersion and decrease its particle size to achieve a good dispersion in the application substrate [2]. In particular, the latent pigment technology [3] was feasible and carried out to reduce a large amount of time and energy consuming of the conventional method.

Zambounis et al. [4–6] firstly developed a latent pigment approach to improve the dispersion of the insoluble parent pigment in paints, plastics, printing inks and so on. The latent pigment is soluble and easily is dispersed in application medium without any time- and energy-consuming treatment. When the latent pigment is used, the protective groups can be removed from the application medium and converted into a parent pigment in situ through proper treatment such as thermal or acid treatment [7,8]. Many researchers have also used the same technology to produce other latent pigments and investigated the pigmentation of their application in several fields. For example, boc-diketopyrrolo pyrrole (boc-DPP), a latent pigment, was treated by a thermal process to obtain the parent pigment, DPP; and a derivative of P.Y 151 was converted into a parent pigment using a heating treatment [9]. Ichimura et al. [10] studied the pigmentation of boc-DPP and boc-indigo through thermal and photo acid-catalysed treatment. An unsubstituted diketopyrrolopyrrol and quinacridone (QA) were used to fabricate field-effect transistors (FETs) by spin coating, offering a low-cost process rather than an expensive vacuum technology [11]. In addition, newly developed film formation technique using latent pigment and laser irradiation gives the excellent film having high transparency, excellent absorption characteristics and high durability [12].

Acid-catalysed deprotection of acid-labile pendant groups, such as *t*-boc and tetrahydropyranyl moieties, was widely applied in positive photo resist [13,14]. The source of the acid species is generated from a photo acid generator (PAG), which is a radiation-sensitive acid generator that generates a strong acid after exposure. *Tert*-butyl ester is a well-known thermal cleavage resin, which

* Corresponding author. Tel.: +886 4 22840510#709; fax: +886 4 22871787.
E-mail address: wtcheng@dragon.nchu.edu.tw (W.-T. Cheng).

http://dx.doi.org/10.1016/j.dyepig.2014.01.021
0143-7208/

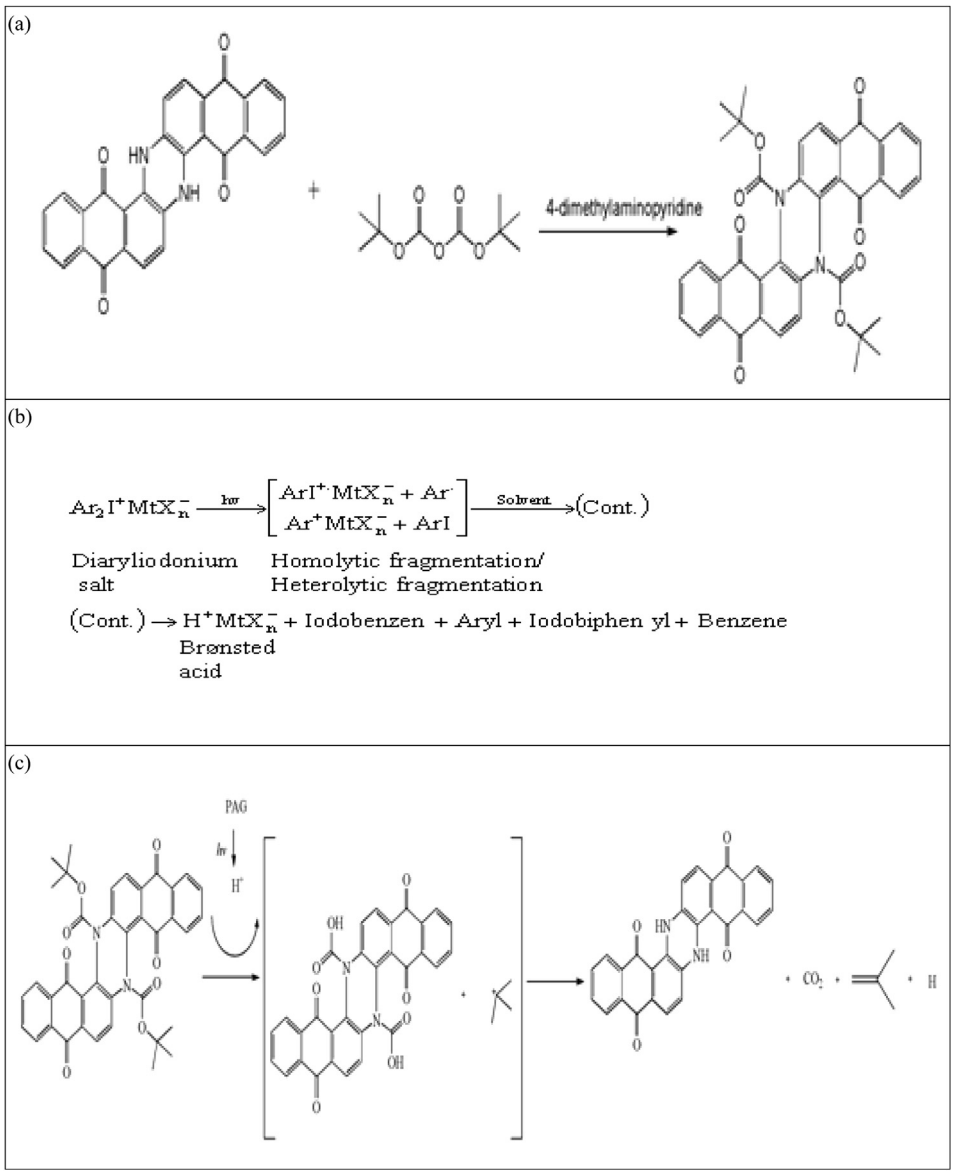

Fig. 1. Schematic mechanisms of (a) the synthesis of boc-indanthrone, (b) the photolysis of PAG (CD-1012), and (c) the conversion of boc-indanyhrone by acidolytic treatment.

thermal reaction normally occurs well above 150 °C. However, in the presence of a trace acid, the thermolysis temperature can be reduced to below 100 °C [15,16].

Although a number of latent pigments have been synthesized, and their applications through thermal or acid treatment in many fields have been accomplished for the crystal phase, a few literature reported the particle size and morphology of the regenerated pigment converted from the latent pigment. Additionally, the crystal phase affects the thermal stability, solubility, hue and others properties of pigment [17]. Therefore, the effects of conversion condition on the crystal phase, particle size and morphology of the regenerated pigment by acid treatment were investigated in this study. This research focused on differentiating the crystal structure and morphology of regenerated pigment converted from latent pigment by acidolytic treatment in organic solvent. To address the shortfall, a well-known high-performance pigment, namely, indanthrone, was chosen as a model compound in this investigation. To improve the solubility of indanthrone in solvent, boc-indanthrone was synthesized by replacing the H atom of the NH group on indanthrone with a compound

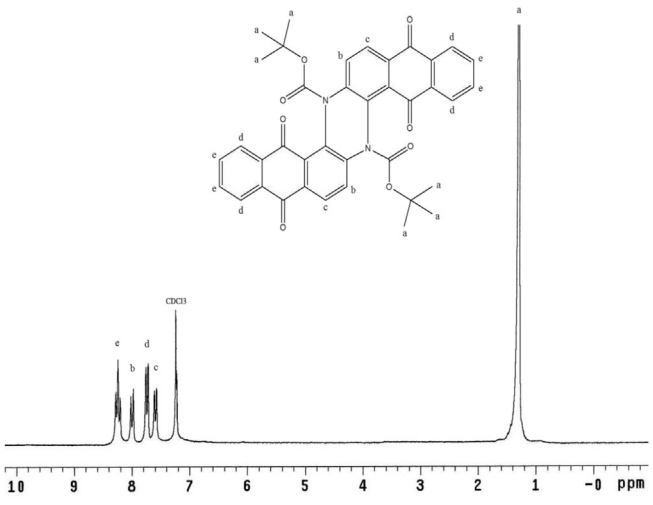

Fig. 2. ^{1}H NMR spectrum of boc-indanthrone synthesized in this study.