

Grinding or What? A Proposal for Alternative Pigment Manufacture

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Abstract

Printing relies heavily on dye pigments for color ink-jet, electrophotographic, and laser printing. Presently, almost all pigments used are chemically produced in bulk and subsequently grind to the desired size. This leads to crystal fragments of uncontrolled morphology and size distribution. The grinding process itself is inefficient in energy consumption and in yield of the desired product.

The author proposes to replace grinding by a crystallization process that tightly controls the morphology of the pigment crystals and their size distribution. Control of size and morphology will yield ultimate control of the color and hue of the pigments and will allow adjusting these parameters at will. Crystallization might preferentially be controlled in the chemical process of dye formation. Alternatively, controlled precipitations may be performed after the dye has been purified, or during a recrystallization procedure. A difficulty for this procedure is the control of the nucleation and growth processes during precipitation of the materials.

Precipitation of insoluble materials is defined by a relatively short time in which the crystals are formed (nucleation) followed by growth. Of these, nucleation is more difficult to control and is more important for the control the final crystal size and product yield.

Significant experimental and conceptual contributions have been made to model the control of the nucleation process, both for batch and for continuous precipitations.¹ These concepts and models are generally applicable for controlled precipitation of organic and inorganic materials.

Of particular interest for the production pigments and dyes is the application of these concepts to the controlled manufacture of these materials. The controlled precipitation process can provide greater control of crystal size and morphology, and savings in the manufacturing process.

An overview of modern techniques and models for the control of crystallization will be presented. This review concentrates on the progress in modeling the nucleation process of particles by a balanced nucleation-growth (BNG) process.¹ The BNG model will be compared with other models that try to predict material nucleation. Compared to other models, the BNG model allows one to quantify the nucleation rate, maximum growth rate, and supersaturation during the nucleation period as a function of nucleation efficiency and maximum growth rate of the crystals. From this model, equations are derived that correlate the number of stable crystals formed with molar addition rate of reactants, solubility of the crystals, and temperature. The BNG model predicts the experimental result that many crystallization processes result in a limited number of crystals followed by growth. The model also predicts that factors like diffusion and kinetically controlled growth processes, Ostwald ripening agents, and growth restrainers control the crystal number. Equations are given for each of the variables that agree with experiment. The BNG model predicts the conditions for renucleation (formation of new crystals during precipitation). It leads to new equations for the prediction of crystal number and crystal size during controlled continuous precipitation in the continuous stirred tank reactor (CSTR) as a function of precipitation conditions.

Table 1: Comparison of Crystallization Models for Batch Precipitations

Models				Modeled Effects
BNG ¹	Classical ²	K&M ^{3,4}	Primitive ¹	
Y	N	Y	N	Crystal Number & Size ^{4,5}
Y	N	Y	N	Temperature ^{4,5}
Y	N	Y	Y	Addition Rate ^{4,5,6}
Y	N	Y	N	Solubility ^{4,5}
Y	N	N	N	Diffusion Controlled Growth ^{4,7}
Y	N	N	N	Kinetically Controlled Growth ⁷
Y	N	N	N	Ripeners ⁸
Y	N	N	N	Growth Restrainers ⁹
Y	N	N	N	Renucleation ¹⁰
Y	N	N	N	Morphology Change ¹¹

Table 2. Comparison of Crystallation Models for Controlled Continuous Precipitations

Models		Modeled Effects
BNG ¹	R-L ^{13,14}	
Y	N	Crystal Size
Y	Y	Residence Time
Y	N	Addition Rate
Y	N	Solubility
Y	N	Temperature
Y	N	Maximum Growth Rate
Y	N	Supersaturation
Y	N	Supersaturation Ratio
Y	N	Nucleation/Growth Ratio
Y	N	Critical Crystal Size
Y	N	Nascent Crystal Size
N	Y	Crystal Size Distribution

In Tables 1 and 2, the range of application of the BNG and other models is compared for controlled batch and continuous precipitations. Note that the BNG model covers both processes while previously only unrelated models were available.

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Biography

Dr. Ingo H. Leubner received his Ph.D. in physical chemistry with minors in inorganic, organic, and chemical engineering from the Technical University (TU) in Munich, Germany. After a post-doctoral fellowship at Texas Christian University in Fort Worth, Texas, he joined Eastman Kodak Company. A significant part of his studies at the TU and at Kodak was the correlation between structure, color, and photochemistry of dyes.

Another aspect was his work on the controlled precipitation of silver halides to control crystal size, morphology, and photosensitivity. In addition to fundamental studies he worked on product development, leading teams to provide products with significantly improved features. His interest as a consultant and speaker is to apply his knowledge in photographic and imaging science, and precipitation to help solve industrial challenges and to introduce the concepts to academic teaching institutions.