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An investigation of the acoustic emission generated during crystallization process of salicylic acid

XingJun Wang [a,b,*], Ying Huang [c]

[a] Qiannan Normal College for Nationalities, Department of Physics, LongShan Road, 558000 Duyun, PR China
[b] Ecole des Mines de Saint-Etienne, SPIN, CNRS 5307, LGF, F-42023 Saint-Etienne, France
[c] Taizhou University, Collaborative Innovation Center, Shifu Road, 318000 Taizhou, PR China

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ABSTRACT

Due to its non-invasive and non-destructive nature, Acoustic Emission (AE) has been successfully used in a wide variety of solids elaboration process. But few papers reported that solution crystallization process give rise to acoustic emission signals that could be related to the basic crystallization phenomena. This study is intended to evaluate the potential for using acoustic emission to monitor the physicochemical crystallization process. The potential relationship between the generation of acoustic signals and the physicochemical crystallization process of salicylic acid (SA) is analyzed by intensity of AE signal and pH value of the solution. Due to its great impact on end-product quality, SA crystal particles are investigated under the influence of injection rates. The results demonstrate that the mode of the particle size distribution is inversely related to the injection rates. In addition, the intensity of AE is obviously influenced by the injection rates. Moreover, the changing tendencies of AE are related to the basic crystallization phenomena, and the intensity of AE can be an important technical parameter to characterize the physicochemical crystallization process. The valuable AE information could increase process understanding and provide a basis for innovative online monitoring and control application.

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1. Introduction

It is reported that over 90% of all active pharmaceutical ingredients (API) would go through a crystallization step at some stage of their production cycles [1]. As other high value-added chemical unit operations in pharmaceutical industry, crystallization, as a main unit operation strongly affecting the end-product characteristics, requires to be monitored during time. Crystallization is a complex process and many parameters may affect the process of crystallization and the final quality of products such as, solution concentration, standing time, solvent–anti-solvent ratio, temperature, stirring speed and solution injection rate. Apart from the reacting conditions, the properties of particulates, especially particle size distribution is considered to be one of the most critical quality-affecting attributes.

The traditional method for particle size detection includes screening method, microscope method, sedimentation method, and inductive method. In recent years, advanced detection technology has been developed rapidly, and it has widened the detection range of particle size. There are Focused Beam Reflectance Measurement (FBRM) [2–3], laser diffraction [4–5], mass spectrometry [6] and pressure fluctuations [7]. Due to its simple, low cost, no special requirements, screening method is often used in industry for particle size measurement. The microscope method is suitable for the measurement of fine particles, and requires the clean test environment. However due to the unsteady-state dynamic features of the crystallization performed by using batch processing, these techniques are not only relatively complex to apply, but also relatively difficult to interpret. Moreover, a lot of chemical production processes are often carried out in hard process conditions, for example in high pressure and high temperature conditions, corrosive media and dust content. As a result, until today it is still difficult to do on-line detection and controlling particle size as all existing technologies exhibit major limitations depending on the basic feature of the slurry, properties of the particles.

As far as crystallization control is concerned, there is a need to develop reliable monitoring techniques that can be used for on-line detection. Although several methodologies are suggested to monitor the crystallization process, e.g. differential scanning calorimetry (DSC), pulsed nuclear magnetic resonance (pNMR), X-ray diffraction (XRD), rheology and polarized light microscopy (PLM) [8], only two of them can actually monitor microstructural characteristic. Furthermore, all these techniques are applied off-line in the assumption that the tested batch is the same as the entire production. Therefore, developing a simple, rapid and accurate on-line detection technology to monitor the crystallization process and particle size is of great significance for quality control.

* Corresponding author.
E-mail address: xingjun.wang@gmail.com (X. Wang).

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Acoustic measurement technique, based on vibration energy produced during manufacturing, processing or material transport, has shown potential as a basis for the development of on-line monitoring and control systems. It has been applied in various areas of research and industrial process monitoring, such as tablets [9], slurries [10], powder blending [11], heterogeneous reactions [12–13], various fluidized bed processes [14–17], and end-point detection in high shear granulation [18–21]. However, few studies deal with the AE monitoring of crystallization processes [22–24].

Acoustic monitoring techniques, in comparison to optical techniques such as spatial filtering technique, near infrared and image analysis [25–30], do not require a window or port into the process vessel. So, there is no need for equipment modifications and it can avoid the inaccurate measurements or impossible of data collection caused by fouling optical probe head or window. Thanks to the large amount of collected data, one can explore a new path for gaining a new point of view on basic crystallization phenomena, to increase process understanding and provide a basis for innovative online monitoring and control application.

This work aims at evaluating the potential for using acoustic emission to monitor the physicochemical crystallization process. With this objective, the chemical reaction of sodium salicylate with sulfuric acid was selected as a model-system. The acoustic waves emitted by the process under consideration can be characterized by an important number of parameters which, roughly speaking, are related to the frequency of the acoustic bursts. Multivariate data were collected and try to find the relationship between the stages of production of salicylic acid particles and try to characterize the basic crystallization phenomena. The intensities (the counts) of AE signals and their relationships with the mode of the particles distribution were analyzed at five different injection rates.

2. Materials and methods

2.1. Materials

Salicylic acid (SA) is known for its ability to ease aches, pains and reduce fevers and it is commonly found in its prodrug form of aspirin (acetylsalicylic acid). Salicylic acid is probably best known as a key ingredient in many skin-care products as with other hydroxyl acids. It has a very low solubility in water but is highly soluble in a wide range of organic solvents e.g. methanol, ethanol, and THF.

In this study acoustic emission was recorded during the precipitation of salicylic acid, which was selected as a model system. Salicylic acid was prepared thanks to the semicontinuous acidification of sodium salicylate solution by sulfuric acid (H2SO4). The chemical reaction is:

$$2RNa + H_2SO_4 = 2RH + Na_2SO_4 \quad (1)$$

2.2. Experimental apparatus and procedure

A schematic of the crystallization setup used during the present study is displayed in Fig. 1. Two flat-bottomed and jacketed 1 L glass vessels were connected via a peristaltic pump (Watson 313s) so as to add the solution of sulfuric acid (H2SO4) in glass n°1 into the reactor (glass n°2). Baffles of stainless steel were used to avoid vortex formation and a high efficiency propeller (Mixel TT TM) was set in the crystallizer with a rotational speed of 200 rpm/min across all experiments so as to maintain a good homogeneity of the particles in suspension. The reactor was a cylindrical vessel with a diameter of 0.05 m and maximum height of 0.1 m. The clearance of the agitator from the bottom of the reactor was 0.005 m. The injection point was inside the liquid midpoint between the agitator position at the center and the wall of the reactor.

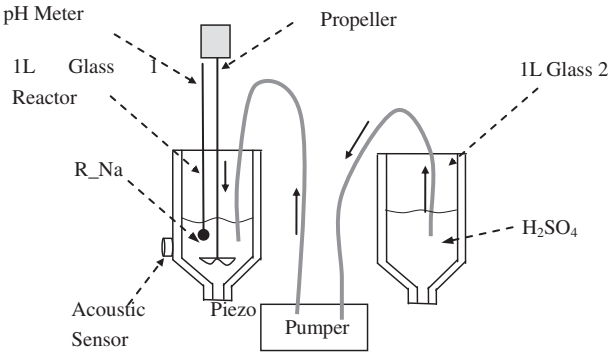

Fig. 1. Experimental setup for the semi-continuous precipitation of salicylic acid.

The AE signal was detected by a sensing device (Physical Acoustics Corporation, WD FS62), and a piezoelectric sensor was fixed on the outside wall of the reactor. Coupling grease was used between the glass wall and the sensor for improving the transmitting efficiency of acoustic signals.

The collected signals by the detecting device were then conditioned, amplified, filtered, and processed with a Data Acquisition System (DAS from EuroPhysical Acoustics S.A). To avoid the disturbance of signals from background, the threshold of detection was set in a high level. The parameters of the AE equipment for our experiment were set as shown in Table 1.

The temperature in the reactor was controlled at 20 °C throughout all experiments during the whole process by manipulating the set-point temperature of a heating bath. The solution of H2SO4 was prepared in reactor n°2 and fed to the agitated solution of sodium salicylate. The concentration of the latter was 0.2 mol/L, it was initially prepared in the glass reactor n°1. The crystallization was initiated during the transformation of solid RH. The rate of reaction was therefore controlled by the injection rate of H2SO4. The injection rates can greatly affect the speed of reaction. In order to avoid the extreme situation, five points are selected randomly in a range between 5 and30. The dilute sulfuric acid was released into the solution of sodium salicylate at five different injection rates of 6.4, 9.6, 16.5, 20.8 and 28 mL/min, to study the effect of injection rate. A peristaltic pump (Watson 313s) was used to vary the injection rate. The whole experimental process was monitored continuously by the AE equipment, which started before the beginning of the introduction of H2SO4, and was stopped when acoustic emission counts or pH value reaches a steady state. pH value was measured by a device (Consort C861) during the whole process. Samples of SA suspensions were withdrawn from both sample points of the crystallizer at the end of the batch processes for off-line size measurements. The particle size distribution analyses were performed using laser diffraction (Mastersizer 3000, Malvern Instruments Ltd., Worcestershire, England). Sample dispersion is controlled by a range of wet dispersion units. These ensure that the particles are delivered at the correct concentration and in a suitable, stable state of dispersion to make accurate and reliable particle size measurements. The glass vessels were washed with sodium

Table 1
Characteristics and set-up parameters for the AE acquisition system.

Instrumentation	Characteristics and setup
DAS unit	AEDSP 32/16-2
Channel number required for the test	1
Threshold(0 dBAE Ref. 1 1 V/sensor)	27
Sensor type:	Physical Acoustics Corporation, WD FS62
Peak frequency (kHz)	541.99
Preamplifier filte (kHz)	20–1200
Preamplifiers (dB)	40
PDT-HDT-HLT(μs)	20-50-120
Max. value (dB)	63.86

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