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Development of a Stereo Imaging System for Three-Dimensional Shape Measurement of Crystals

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Abstract: Despite the availability of various Process Analytical Technologies (PAT) for measuring other particle properties, their inherit limitations for the measurement of crystal shape have been restricted. This has impacted, in turn, on the development and implementation of optimisation, monitoring and control of crystal shape and size distributions within particle formulation and processing systems In recent years, imaging systems have shown to be a very promising PAT technique for the measurement of crystal growth, but still essentially limited as a technique only to provide two-dimensional information. The idea of using two synchronized cameras to obtain 3D crystal shape was mentioned previously (Chem Eng Sci 63(5) 1171-1184, 2008) but no quantitative results were reported. In this paper, a methodology which can directly image the full three-dimensional shape of crystals has been developed. It is based on the mathematical principle that if the two-dimensional images of an object are obtained from two different angles, the full three-dimensional crystal shape can be reconstructed. A proof of concept study has been carried out to demonstrate the potentials in using the system for the three-dimensional measurement of crystals.

Key Words: Stereo imaging, Crystal shape, Three-dimensional shape reconstruction, Image analysis

1. INTRODUCTION

Particle shape is known to be extremely important to many pharmaceuticals, biopharmaceuticals, human health products and speciality chemicals in solid form. In the pharmaceutical industry, morphology can affect important properties such as dry powder density, cohesion, and flowability, which can have major impact on a company's ability to formulate drug particles into finished products. Moreover, crystal morphology can affect drug dissolution, potentially affecting finished product bioavailability and, in extreme, resulting in the loss of the license to making the drug product.

Despite its significant potential importance, the direct characterisation of particle shape has been quite limited largely relying on off-line instruments of methods. For quite some time there have been no effective instruments capable of providing real-time information on particle shape particularly with the capability for use during the processing of particles in unit operations such as crystallisation, precipitation, granulation and milling (dry or wet). Whilst, well-developed and studied PAT (process analytical technology) techniques such as acoustic, and mid and near infrared spectroscopy and laser diffraction have been used in process monitoring these techniques cannot give detailed information on particle shape though some of these techniques have been shown to be able to distinguish between different polymorphs with careful spectral data analysis using chemometrics [1]. Overall, the inability in the measurement of particle shape and growth rates in individual faces has greatly restricted the development and implementation of monitoring and control of particle shape for these particle formulation and processing systems.

Most recently, several research groups and industrial companies have found that it is feasible to use on-line two-dimensional (2D) imaging for particle shape measurement, and initiated research activities [2-23]. Rawlings and co-workers carried out research on the measurement of crystal size and shape distributions using in situ video images [22]. Wilkinson et al. from GlaxoSmithKline has developed a prototype on-line, non-invasive microscopy imaging system [17, 23] for monitoring pharmaceutical crystallisation. AstraZenaca [21] tested the use of a commercially available imaging probe named as PVM by Lasentec [24]. Other 2D imaging systems such as PIA [25] and ISPV [26] are also available. Mazzotti and co-workers [27, 28] designed a flow through microscope with a mirror configuration, hence the passing particles can be viewed from two directions. They applied the system to ascorbic acid crystals for extracting crystal length, width and depth information. Ma et al. [29] used single 2D images, together with crystal shape structure, to obtain the normal

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distances of individual faces to the crystal centre, hence constructing 3D shape, of potash alum crystals in a hot-stage reactor. The evolution of these normal distances over time then leads to the estimation of the face specific growth rates. However, the developed method can only be applied to some crystals with specified shape features and imaging conditions. A flow through microscope system was also used to obtain high quality images with regard to particle sharpness [30]. The boundary curve of the 2D particle projection was used to estimate 3D shape. In medical and biological areas, miniature CCD cameras were used to develop imaging systems. Gorpas et al. [31] developed a volumetric method, using a binocular machine vision system with a structured light projector, to reconstruct three-dimensional (3D) tumour surfaces (ca. 10 mm in size) with an accuracy of 0.04 mm in standard deviation. A imaging system using a flow through cell with one camera and two mirrors was developed to obtain 3D information of crystals grown from solution [32]. Another binocular vision system was designed to reconstruct the shape of mineral and rock particles on a conveyor belt [33]. A few of three cameras imaging systems were also developed to characterise 3D shape of free-falling particles (100μm ~ 4mm) [34] or 3D information of a wound area [35]. However, all these binocular and three-camera imaging systems are not designed to be used in a reactor for direct measurement of crystal size and shape evolution and the objects to be characterised are normally at millimeter scale, hence not being suitable for micron-sized crystals. Interactive systems that allow users to control and manipulate real-world objects within a remote real environment are known as teleoperator or telerobotic systems [36, 37]. Such systems are often used in medical applications to confirm diagnosis and make telepresence surgery. Some 2D/3D endoscopes have been developed for these purposes (see for example [36, 38-45]) with the most known ones being da Vinci telerobotic surgical system [38] and ZEUS™ [39]. Such systems are controlled by surgeons remotely by viewing virtual surgical site with stereoscopic system and controlling stereoscopic camera and robot surgical armaments. In their papers, Hu et al. [41, 42] developed 2D and 3D surgical imaging device with pan and tilt for minimally invasive surgery. However, these imaging systems still have significant limitations in terms of their image processing capabilities and how to link the information for shape monitoring and control. In fact at the moment they are mainly used to display information to operators and store data onto hard drive. Nevertheless, we believe that with the active research activities internationally and the great interest from instrument companies and end-users, before long the image processing capabilities will improve significantly, thus opening up new commercial opportunities for on-line monitoring and control of crystal size distributions based on the shape evolution.

Despite the several research and development programmes internationally, all the work on particle size and shape measurements is restricted to 2D imaging. Although 2D shape is already a major step from the traditional characterisation of particles based only on a volume equivalent diameter of a sphere, being able to measure 3D shape will be of much greater scientific and industrial significance. Some microscopic systems are able to provide 3D information of a particle by scanning many thin sections of the sample [46]. However, these systems are not practical for 3D on-line measurements due to the very low speed in operation. Intensive research and development has been carried out to obtain 3D topographical data of objects in robotics and machine vision areas with the objects at meter or millimeter scale. Several optical, non-contact methods have been developed for a wide range of applications, such as moire methods (shadow and projection), fast-Fourier transform approaches, stereo imaging etc. Stereo imaging has the advantage of providing more direct, unambiguous and quantitative depth information, and it can be used for a very wide range of applications in academic research, industry and daily life in addition to the applications of 3D measurement. Most approaches to the application of stereo vision utilise the human vision system to establish a model for the camera system [47]. For stereo imaging system, many different camera-object geometries have been studied and used for specific applications such as the common parallel camera optical axes, the converging (nonparallel) camera optical axes, etc. To extract 3D information from the recorded stereo image pairs for 3D reconstruction of objects, it is necessary to find disparities among a series of corresponding points between a pair of stereo images taken from the same scene. There are many matching techniques and corresponding algorithms which can be generally divided into three categories: area-based, feature-based, and their combination. In general, feature-based techniques yield a better match more stably and accurately than other techniques. Two types of features commonly used are point-like features such as corners and line segments [48].

In this paper, a proof of concept study is reported to demonstrate the potentials in using the system for the 3D measurements of particles at micron scale. The basic mode of the operation is based on the mathematical principle that if the 2D images of an object are obtained from two different angles, the full 3D particle shape can be recovered. In the following sections, the methodology of 3D stereovision imaging system for the characterisation of crystal shape is briefly described. Then the stereo imaging system is developed with some case studies. Finally, concluding remarks are made.

2. THE METHODOLOGY

Figure 1 shows the development flowchart for the reconstruction of 3D crystal images from 2D stereo images taken by a stereo imaging system. A two-camera system is proposed, which places two cameras at a pre-defined angle (stereo angle). The two cameras focus on the same sample volume with the identical camera parameters. The stereo image pairs can be obtained via these two cameras when shuttering at a synchronised instant. The obtained stereo image frames are processed using a multi-scale segmentation algorithm [4] or other pre-processing methods. Using corner/edge detection [49-51], the corners and edges of the crystals from the processed images can be identified. A feature-based matching algorithm (see for example [48]) can be used to identify the corresponding left and right features (corners, edges). The 3D coordinates of crystal shape can then be reconstructed with the identified corresponding corners or edges using a stereo triangulation algorithm [52, 53]. The obtained 3D crystal shape information can be further used to obtain other useful data. For example, the dynamic change of 3D shape of crystals with time, can be used to estimate crystal face growth rates and derive facet growth kinetics (e.g. facet growth rate as a function of supersaturation).

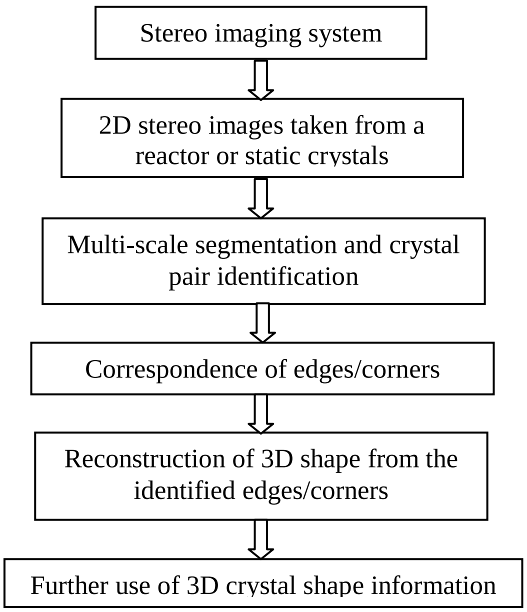

Fig 1. Flowchart of 2D stereo image measurement and 3D crystal shape reconstruction

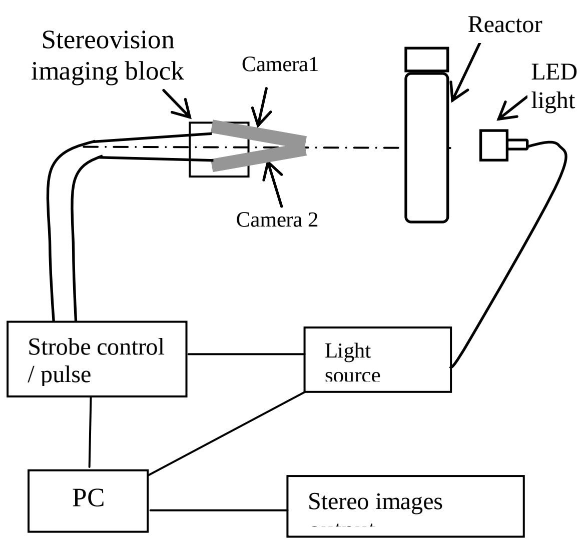

Fig 2. Schematic drawing of the stereovision imaging system

3. EXPERIMENTAL STUDY

The schematic diagram of a stereo imaging system is shown in Figure 2. The system can be composed of a reactor for crystallization processes under a controlled heating and cooling conditions, and a stereovision imaging system for capturing stereo image frames. The recorded stereo image pairs are stored onto a PC for further image analysis and 3D reconstruction.

Technique to use stereo cameras (two cameras) for the reconstruction of 3D vision is directly related to human vision system, i.e. two cameras mimics our left and right eyes and 3D reconstruction software mimics the fast image processing of our brain. The stereovision technique has been widely used in machine vision such as vehicle and person identification, product manufacturing etc. though most of them positioned two cameras in parallel. In chemical processing area, stereovision systems for 3D reconstruction of particles in reactors at micron size scale are still rare because of the technique challenges when the object size is reduced from millimeters or meters to microns. The proposed stereovision 3D imaging system in this paper is to face the challenges and provide a practical tool for 3D reconstruction of micron-sized particles in reactors. A typical built-up stereo imaging system (Figure 3) includes a 3D imaging block (two cameras with two telecentric lenses), a light source, a light controller with strobe pulse for synchronising cameras and lights, a reactor, and a PC with image acquisition software and image processing. The imaging system has a spatial resolution of 3.45 μm with the current selection of cameras and lenses. The system can capture up to 6 frames per second with each image having a resolution of 2448×2050.

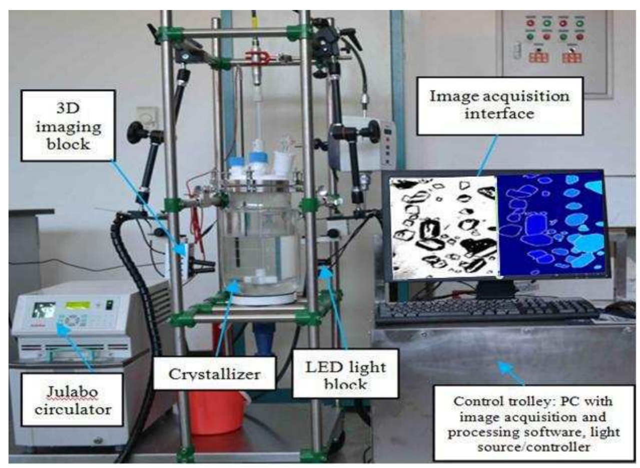

Fig. 3. 3D imaging system

4. 3D SHAPE RECONSTRUCTION FROM 2D STEREO IMAGES

With the recorded image pairs, each image is undertaken several image processing steps including pre-processing such as the adjustment of image contrast, edge/corner extraction, and the identification of edge/corner correspondence for 3D reconstruction. Using the multi-scale segmentation method [4], the crystals from each image of an image pairs can be identified and numbered for further processing to obtain 3D shape information. The central coordinates of the numbered crystals are calculated. The paired crystals between the two images in an image pairs can then be found by comparing the central coordinates of the numbered crystals in each image. The identified crystal pairs will be further processed to reconstruct 3D shape. According to the different features from different crystal shapes such as needle-like, plate-like, rod-like etc., the corner correspondence between the identified crystal pairs can be established. This method can avoid the search of the whole image to obtain the matched corners, hence improving processing speed and quality.

For 3D reconstruction from 2D stereo images, in addition to stereo matching to obtain the correspondence of the identified crystals corners in the paired images taken from the same scene, the reconstruction of an object from the calculated correspondence can be achieved by using the triangulation method [52, 53]. For the 3D coordinates of a corner, the 3D coordinates (X, Y, Z) of this corner is a function of the coordinates of the two 2D images (x_1, y_1, x_2, y_2), stereo angle (α), the total distance (L) between a subject and camera, other properties such as resolution (σ), magnification (Δ) etc.

$$(X, Y, Z) = f(x_1, y_1, x_2, y_2, \alpha, \sigma, \Delta, L, \ldots) \tag{1}$$

In the current imaging configurations of the cameras and lenses, the stereo angle between the two camera optic axes is fixed at 22 degrees and the total distance L including the lens working distance, lens length and camera flange focal distance, has a value of 147.726 mm. The magnification of the lenses, Δ, is 2 times with a resolution σ of 4.65 μm. The obtained equation for the calculation of 3D coordinates of a corner or point from the two 2D images is as follows.

$$\begin{bmatrix} X \\ Y \\ Z \end{bmatrix} = \begin{bmatrix} a_{11} & 0 & 0 \\ 0 & a_{22} & 0 \\ 0 & 0 & a_{33} \end{bmatrix} \begin{bmatrix} b_1 \\ b_2 \\ b_3 \end{bmatrix} \tag{2}$$

where

$$\begin{bmatrix} b_1 \\ b_2 \\ b_3 \end{bmatrix} = \begin{bmatrix} \frac{\sin\frac{\alpha}{2}}{\sin\alpha} \\ 1 \\ \frac{\cos\frac{\alpha}{2}}{\cos\alpha} \end{bmatrix} \tag{3}$$

$$\begin{bmatrix} a_{11} \\ a_{22} \\ a_{33} \end{bmatrix} = \begin{bmatrix} a_{10} + (x_1 + x_2)\frac{\sigma}{\Delta} \\ y_1 \frac{\sigma}{\Delta} \\ a_{30} + (x_2 - x_1)\frac{\sigma}{\Delta} \end{bmatrix} \tag{4}$$

with a_{10} = 74 mm and a_{30} = 54.5 mm. The reconstructed 3D particles are then used to calculate particle shape descriptors and also perform classification and clustering of particles.

For a real needle-like crystal (a line segment in theory) as shown in Figure 4(a), the real length of the line is 1000 μm. However, it can only measure as 1000 μm on a 2D image when the line is at an orientation perpendicular to the camera optic axis. In the current case, the line has a length of 850 μm on the 2D image, which indicates that the line has an angle of ~60 degree against the optic axis. With the crystal pair identification and edge/corner detection, the coordinates of the end points for the paired lines on the 2D image (for example, Figure 4(b)) can be used to reconstruct the 3D coordinates of the end points of the line, hence the line in real space (Figure 4(c)). The reconstructed 3D line has a length of 991 μm, which is about 1% from the real length of 1000 μm. It can be seen that it will present significant error if the 2D length of 850 μm (representing a 15% error of the real line length of 1000 μm) for this line is used for further crystal characterisation. Furthermore, at the extreme configuration of a line that parallels to the camera optic axis, the line length on a 2D image will be close to zero.

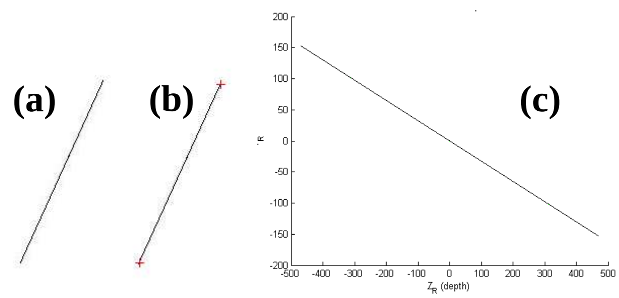

Fig 4. Reconstruction of a line with a length of 1000microns: (a) 2D image of the line; (b) identified end points of the line; (c) reconstructed line in 3D space

For the image pairs captured from a plate-like crystal in a reactor (Figure 5(a)), the application of the multi-scale segmentation method and the crystal pairs identification procedure will identify the crystal pairs (Figure 5(b)) for further processing. Based on the features of a plate-like crystal, a parallel fitting procedure is applied to the identified crystal pairs to obtain the 2D coordinates of the corresponding corners (Figure 5(c)). Again, due to the plate-like crystal is not orientated perpendicular to the camera optic axis, the reconstructed crystal (Figure 5(d)) has larger length and width, hence larger area comparing to those from pure 2D calculations.

For an ideal crystal of L-glutamic acid (LGA) α-form, the same crystal pair identification procedure can be applied to the original 2D images (Figure 6(a)) and the identified crystal pairs were then processed with edge/corner detection and correspondence search. The reconstructed 3D shape of the LGA α-form crystal is shown in Figure 6(b). Note that with the help of crystal symmetrical features, the whole crystal shape of LGA α-form can be established from the reconstructed 3D shape which only represents a half of the real crystal in 3D space.

The typical stereo images of a sugar crystal are shown in Figure 7(a). After performing crystal pair identification, edge/corner detection and correspondence of the left and right images, the coordinates of the corresponding corners on left and right images were used to obtain the 3D shape of a sugar crystal (Figure 7(b)). Again, the crystal symmetry can help to establish the whole 3D structure.

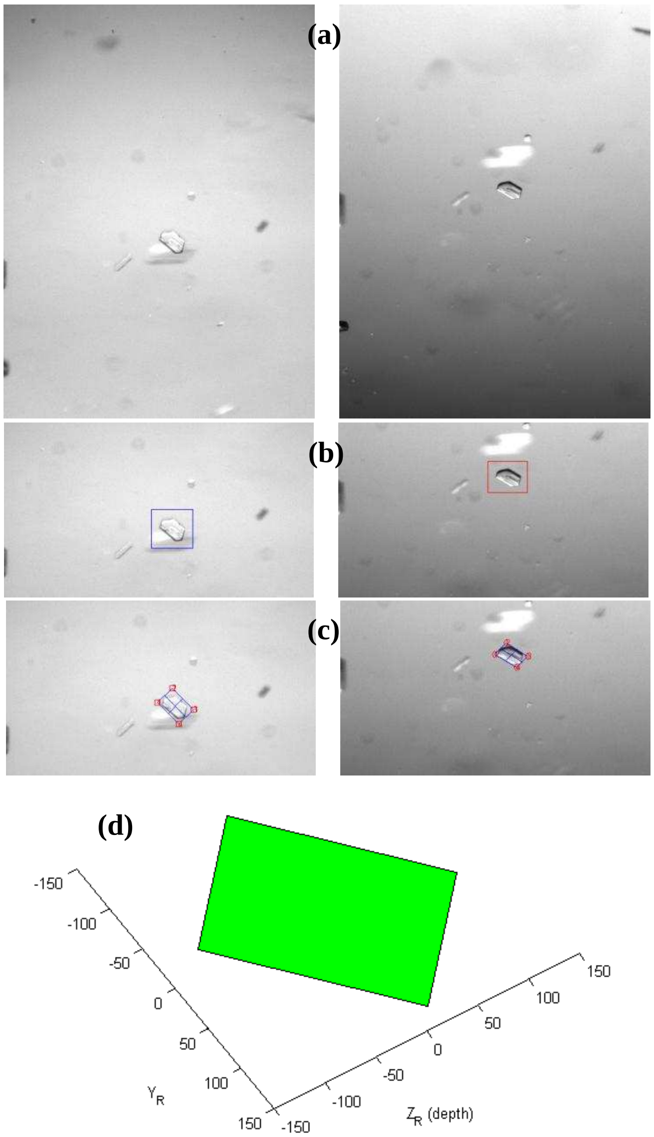

Fig 5. Reconstruction of a plate-like crystal grown from solution in a reactor: (a) original image pairs; (b) identified crystal pairs; (c) feature-based edge/corner detection and correspondence; (d) reconstructed plate-like crystal in 3D space

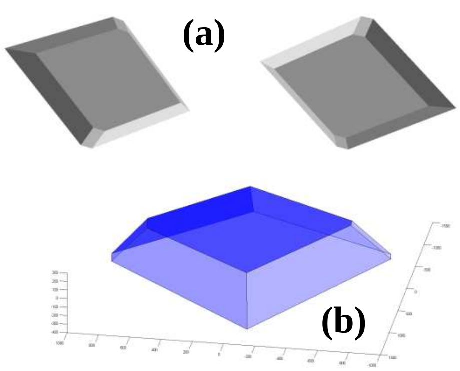

Fig 6. Reconstruction of a crystal of L-glutamic acid alpha form: (a) original 2D images; (b) reconstructed crystal in 3D space

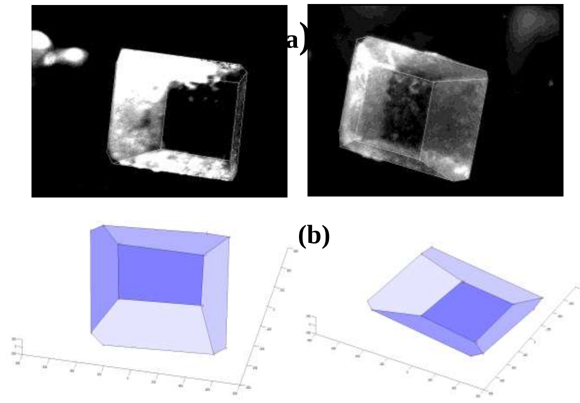

Fig 7. (a) Typical stereo image pairs of a sugar crystal, (b) reconstructed shape of a sugar crystal at two orientations

The typical images of potash alum crystals are illustrated in Figure 8(a) (left and right images). Using crystal pair identification and edge/corner detection with some false corners were manually removed. The corners for correspondence processing were obtained as shown in Figure 8(b). With the corner coordinates, stereo triangulation can be used to calculate the corresponding 3D coordinates. After that, the 3D shape of the crystal was reconstructed as shown in Figure 9(b). For comparison purpose, the nine surfaces of the potash alum crystal in the 2D image were shown in Figure 9(a), each associated with a letter from A to I. The crystal size in x, y and z directions were estimated from the 3D coordinates as 580, 588 and 277μm, respectively.

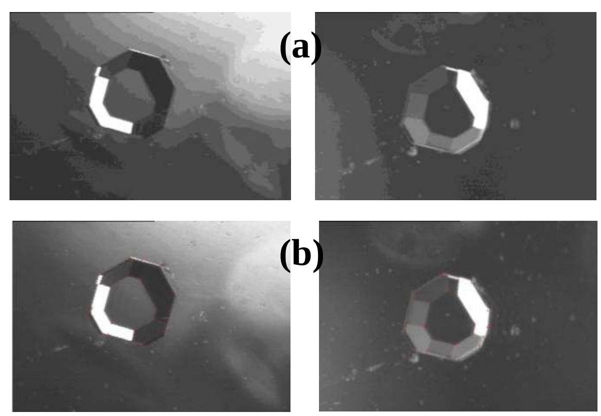

Fig 8. Image pairs of a potash alum crystal (a) original images (b) corner detection

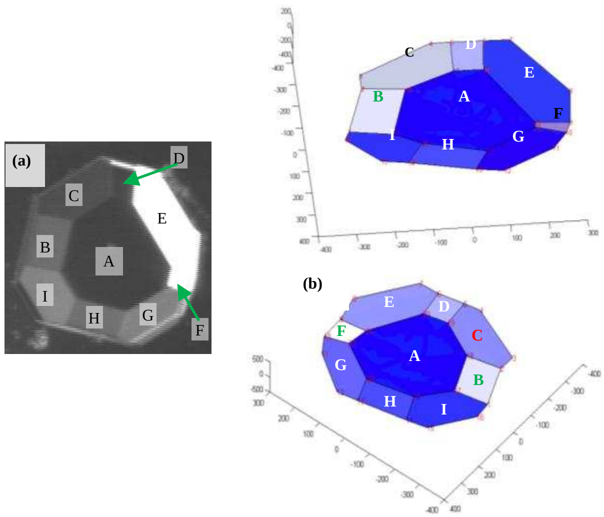

Fig 9. Individual faces of a potash alum crystal: (a) crystal on a 2D image; (b) reconstructed 3D crystal with two orientations (Nine surfaces on the top part of the crystal: one {100} face – A; four {110} faces – B, D, F, H; and four {111} faces – C, E, G, I)

5. CONLUDING REMARKS

Most of the existing imaging systems can only record 2D images and some 3D systems in machine vision and medical applications are only suitable for large objects and/or the visualisation of 3D images as an assistant to remote micro-surgery. The 3D stereovision imaging system presented in this paper is particularly designed to record and reconstruct 3D shape of particles at micron size scale in reactors. The technique uses two cameras and two telecentric lenses to image different facets of a particle, and then to reconstruct the 3D shape. The reconstruction of 3D crystal shape from 2D stereo images can involve the following steps: raw stereo image pre-processing; corner/edge detection; correspondence establishment and triangulation for 3D calculations. The obtained 3D crystal shape can be used to characterise crystal properties such as face growth rates, shape factor, surface area, volume and size distribution. The case studies demonstrated that the instrument, together with the developed methodology, is potentially very useful for process and product quality monitoring and control of crystallisation processes in academic research and industrial applications including pharmaceuticals, agro-chemicals, dyes and pigments, food, detergents and formulation additives.

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