Research Article

Imcube: A Static Particle Size Analyzer for All Shape Types via Advanced Vision Tools and Integrated Machinery

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Abstract

An integrated machinery to perform the analysis of particle size distribution through image processing formulations is presented. The product is comprehensive and flexible to many different industrial needs with a tailored hardware integration and design, which employs a set of sophisticated algorithms for the computational efficiency and accuracy. Compared to the traditional methods, the architecture is superior and provides significant impact as irregular and noncircular particles from a wide dimension spectrum can be analysed instantly, eliminating the need for significant manual effort with conventional trays with low accuracy. The reports are obtained through the built-in screen, mounted on the device, at customer specified detail level in addition to state-of-the-art presentations benefiting common statistics. The prediction performance, which is validated through industrial data, can further be developed for smaller particles, as a higher resolution camera implementation is necessary, with a heuristic algorithm to estimate the maximum likelihood of particle sizes when they overlap on the measurement tray.

Keywords: Image Processing, catalyst, particle size, particle size distribution

1. Introduction

Structure-sensitivity has a great impact on catalytic activities in many industrial reactions. The basic knowledge on catalyst science dictates the reasons for the structure effects in metal-based catalysts as: (i) electronic, (ii) geometric, (iii) strain and compression aspects. The separation between electronic levels is given for N electrons as
δ~εF (Fermi energy) / N. If δ is bigger than 2kT, the separation is observed in the levels, and this phenomenon is called the quantum size effect. The Fermi energy is generally 5eV for metals so the level spacing is 50meV at standard conditions. Hence, the critical size for the quantum size effect would be εF/2kT, ~100 atoms. When the metal particle size changes, this would lead to a change in the atom’s mean coordination number, i.e., as smaller the particles, the higher the number ratio of surface atoms to bulk atoms. Then, the valence band of metal gets narrower, the density of states at the Fermi level decreases (i.e., more localized e-), and the maximum of the valence band and the core level shift towards higher energies. This phenomenon might be the base of the structure effects, possibly more than a quantum size effect. It was supported by Gan et.al. (2001) that Pt nanoparticle below 2 nm size shows non-metallic behavior[1]. Additionally, Englisch et.al. (1997) studied crotonaldehyde hydrogenation and observed an increment in unsaturated alcohol with increasing Pt-metal particle size, but a decrement in metal particle size led to hydrogenation of the olefinic double bond[2]. This finding can be explained by that the electron density of d-orbitals decreases with decreasing particle size, in order that a decrement in repulsive four electron interaction between the metal surface and C=C bond is observed. Moreover, the number of edges and corners of the particle increases with decreasing particle size, leading to free adsorption of double bonds existing in crotonaldehyde. This example explains how particle or crystal size affects the selectivity and turnover frequencies. Additionally, the reactions such as Fischer-Tropsch reactions, carbon formation, and sulfur passivation are highly structure-sensitive reactions [3]–[5]. Ralston et. al. (2017) investigated the carbon coverage with respect to the particle size of synthesized cobalt catalysts by using the chemical transient kinetic system and they found that increment in particle size yielded higher carbon coverage due to the loss of specific B5 types of sites in the catalyst which has smaller particle size. Yarulin et.al.(2012) studied the structure-sensitivity of acetylene hydrogenation over Pd catalysts with different shapes and sizes (e.g., cubic (10 nm), cuboctahedral (4.5 nm), and octahedral (bimodal PSD)). The authors found that the atoms located on Pd 111 and Pd 100 faces yielded the highest activity compared to the atoms on particle edges. Also, the catalyst with an octahedral shape yielded the highest activity while the catalyst with a cubic shape yielded the lowest. Moreover, it was found that the size closer to 2 nm yielded the maximum activity and then, showed decrement with increasing particle size to 20 nm. Turnover Frequency showed an increment between 2-10 nm. Furthermore, Rautanen et.al. (2001) proposed the mechanism for naphthalene hydrogenation over Ni/Al2O3 catalyst. The reaction began with cis addition of two dissociatively adsorbed hydrogen to form tetralin followed by
hydrogenation to hexahydronaphthalene. The observation of a high amount of cis-decalin showed that hexahydronaphthalene was hydrogenated to 9,10-octalin, not to 1,9-octalin and then, it isomerized to 1,9-octalin which further be hydrogenated to cis- and trans-decalin. In addition, the adsorption of naphthalene occurred at one active metal, so naphthalene hydrogenation was structure-insensitive[8] but the adsorption of tetralin formed during the hydrogenation of naphthalene, as depicted in the mechanism by Rautanen et.al. (2002), needed an ensemble of atoms of the active metal and its hydrogenation was structure-sensitive. To show the structure sensitivity in carbon formation, Ni-based catalyst could be a good example that the carbon deposition is difficult on Ni(111) when compared to Ni(100) or Ni(110). To inhibit the carbon formation rate, the control of the size of metals on the surface can be performed by doping because according to Maxted model, the ensembles necessary for carbon formation is relatively large (6-7 atoms).

As it can be seen from the literature, the structure-sensitivity has an intense effect on various catalytic processes. In our refinery, there are many catalytic reactions that are naturally structure-sensitive. Therefore, the particle and crystal size have significant importance throughout many catalyst properties in the manner of operational efficiency. The catalysts whose activities have decreased, are regenarated for the reusability and process intensification to reduce the economic and environmental impact. The catalysts particularly used in the reactors, fractures due to collisions, reducing their particle sizes, and therewith, catalytic activity and reactor performance are affected due to the increment in pressure drop. Moreover, downstream processes are also affected by equipment fouling due to the reduced catalytic activity and accumulation.

The regeneration process is only applied to the catalysts, which are in the appropriate size range. The fast and accurate particle size measurements have great importance especially in catalyst renewal periods to enable the go/no-go decisions. The name-brand equipment in quality control laboratories cannot be used due to the size of the catalysts especially for structured formed catalysts, which clogged the interconnect lines of the analyzer. Alternatively, the sieve method has been used to perform such analysis, but it delivers significant errors especially due to the non-spherical particle shape of the catalysts. Thus, a new approach would give new insight into these conventional laboratory techniques to speed up the analysis process and get accurate results for the catalysts in specific particle size. Therefore, an in-house developed technology called “Imcube®” is designed and fabricated to measure the particle sizes and distributions of the structured catalysts throughout the refinery and petrochemical complex.
Recent advancements in hardware and superior software packages enable the utilization of high-resolution images at high sampling rate. However, those algorithms should be tailored well for practical purposes as those devices are usually designed to perform analysis of particles from different fields of engineering, extending its practicality and application areas for economic considerations. Major engineering limitations are composed of non-uniform and non-spherical particles, which hinder the analysis through traditional methods, overlapping or particles at small scales, and user-interface issues to help keep track of historical measurements and provide a descriptive and simple-to-use analysis report at ease.

With sophisticated developments in the field, image processing has gained significant attention in the last decades despite studies have a longer history [10]. In theory, image processing is an advanced filtering formulation based on convenient mathematical architectures[11] [12] to favor detection and estimation of certain patterns or abnormalities in an image which can be converted to a numerical scheme through various transformations [13]. In addition several image processing architectures are available through open source or commercial software [14].

The Imcube® is a digital solution to architectural and fundamental issues to provide significant superiorities for both user satisfaction and experimental accuracy considerations. The Imcube® uses an in-house developed algorithm through image processing technology, which works with high accuracy and low uncertainty. The resulting high-accurate quantitative data become an important decision-making tool for process intensification. The particle size distributions of fresh and used catalysts from several reactors in the refinery are currently being evaluated and reported via the device. In this study, the conceptual algorithm approach and representative results of the Imcube® will be summarized.

2. Materials and Methods

2.1. Design of the device

The Imcube® has a solid and curled-corner design to reflect a dynamic appearance to its users. The device serves an integrated compact cubic design with a small space requirement. It is suitable for bench-top usage with the size of 34(w)× 54.5(h)× 36(d) cm. The Imcube® is produced via additive manufacturing technology to give flexibility, practicality, and easy-to-modify functionality. In the production of the device, PA12 for multi-jet fusion (MJF) technology is used.
The design of the device is of set purpose to lower the random errors and boosts the precision and accuracy. For this aim, several design considerations were taken into account such as the hindrance of overshadowing, a well-calibrated camera angle and height, and vibrating sample tray in order to set the particles in a thermodynamically favorable position. The Imcube® is a unique device for high-precision analysis at a relatively low cost. Such a sophisticated integration is obtained through advanced image processing algorithms tailored for the calculation of shape-related properties although overlapping particles on the tray. Therefore, the Imcube® delivers quicker and more accurate results, which have been validated by the catalyst vendor data. A high-precision analysis starts with the vibration of the particles, and then, goes on through a user-friendly graphical user interface at front-end, using some mathematical tools at the back end to infer the overlapping regions and shape of the particles to measure the particle size of solids ranging from 100 µm to 10 mm. The back end of the software comprises the detailed particle theory to provide extensive report options to its users.

2.2. Computational Environment

Imcube® benefits from a comprehensive set of image processing algorithms whose tasks are decomposed into different computational architectures for computational efficiency in addition to desired accuracy. As the computational load is handled through sophisticated mathematical formulations to process large-scale images, the current technology is limited by the camera resolution and can be upgraded easily with no significant change in the software algorithms and architecture.

The algorithm can process different shape characteristics including non-uniform and non-circular particles which are defined by user through UI, although common and
globally desired statistics are calculated and reported typically at each analysis. A set of common geometric architectures are introduced, however, to account for the compatibility of a particle to pre-defined shape [15] [16].

There are several segmentation algorithms to benefit from the structural properties of the actual particles despite their overlapping in the analysis tray, despite automated vibration of the tray [17]. With significant number of image segmentation algorithm capabilities, Imcube® benefits from watershed algorithm [18] most, since highest accuracy results under current machinery design are obtained for a wide range of particles. However, the algorithm can process different algorithms simultaneously, sequentially or coordinately, to achieve the best outcome under computationally efficiency considerations. The default implementation covers a sequential and localized watershed algorithm constrained by the maximum and the minimum length of the particles to be analyzed. Furthermore, rather than a global scale, a localized watershed algorithm is implemented sequentially until the particles are segmented through successive and iterative tasks until statistically the best coverage of estimated and observed particles are obtained. In turn, such calculations deliver high accuracy and can be transformed into the analysis of any kind, which can also be tuned for customer required tasks.

3. Result

A typical analysis is performed for demonstration purposes based on actual catalyst from the plant to evaluate the deactivation and probable performance drops in the catalyst. Such an insight is inferable from the catalyst particle size distribution as it plays a major role in the determination of overall catalyst activity and performance due to representation of structural deformation. In practice, with operations at high temperature and pressure, the catalyst deformation fastens and proposed quantitative analysis enable the collection of large amount of data in the long run to estimate the major components of such deformation. Fig. 1 includes raw measurements with a pre-processing on the image for succeeding advanced computations.
Fig. 1 includes the raw image obtained from the camera, although some portion of the image is cropped for demonstration purposes as the catalyst under interest is a relatively small material with high amount of particles. Fig. 2b is a zoomed scale of Fig. 1a to a particular randomly selected region, to better illustrate the impact of overlapping and contact of the particles, which hinder the easy-to-process algorithms to extract knowledge at desired accuracy, in addition to computational concerns. Fig. 2 is obtained after the aforementioned image processing to yield the distribution analysis.

Fig. 2a includes the cumulative distribution of large and small particle distributions based on their equivalent sizes based on sphericity, which is a common measure to report and analyse the particle size distribution, regardless of the actual shape. Based on distribution, F10, F12, and F13 are calculated to account for the surface area and the
volumes of the particles, which are commonly used statistical quantities for the evaluation. Histograms of variables presented in Fig. 2b are shown in Fig. 3.

4. Discussion and Conclusion

Image processing tools for the particle size distribution is an important and promising aspect of next generation laboratory analysis for the estimation of product properties and performance based on easily-measurable methods. Imcube® is tailored to answer those needs, in particular, through employing open-source and sophisticated mathematical formulations to address customer specific tasks as well as common and traditional problems to deliver a user-friendly experience.

The device includes well-tuned hardware specifications in addition to design considerations to account for user-experience for the real-time applicability. For instance, the screen, for a quick-analysis investigation and the device management, is tilted to ensure the orthogonality in eye contact once the device is located on a typical laboratory bench. In addition, remote management capability through internet access enables the diagnostics and updates online in addition to algorithmic developments on specific customer needs. Additionally, the device is composed of common and available hardware to address the upgrades and machinery failures for a fast customer support. In practice, the detail and the intensity of the analysis, as well as the minimum particle size to be detected, depends on the camera which can be upgraded easily once a need arises. In our case, a further detail in analysis at a smaller scale is not needed and left a future work for development. With significant improvements which provide accuracy and computational advancements, several user-experience and user-interface superiorities
are also implement. For instance, self-vibration tray contributes the distribution, increasing the accuracy of the image processing and reducing the computational load for the analysis. Automatized door and screen-controlled lightning delivers a better user-oriented solution at manageable cost, compared to competitive products. Open-source architecture development of the product serves significant superiorities both in terms of market advantage and customer-oriented product development. A major superiority of the approach is the capability to process non-spherical and irregular particles which cannot be processed in common vibrational trays with structured spacing, serving the spherical particles only at desired accuracy. In addition, those are limited to a number of trays, mostly, and requires significant experimental effort as each tray should be seized individually after the analysis is performed. In contrast, the product employs a simultaneous solution to address those shape variability and deliver a continuous spectrum of product analysis, which can be decomposed into desired analysis levels, including the most common ones as in Fig. 3.

References


