HIGH PRESSURE FLOW CELL FOR PARTICLE SIZE DISTRIBUTION ANALYSIS

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ABSTRACT

The high pressure flow cell and industrial vision system provides on-line 2-dimensional particle size analysis for polymer beads. This paper covers required vision system features allowing common imaging sensors to function as an in line particle analyzer. The study conducted demonstrates the system's capacity to obtain particle size distributions that can be used for process control or quality assurance with synthetic polymer beads. The particle size distribution is obtained within seconds and immediately updated to allow for process adjustments. Results are then provided as a graphical computer generated display or output as a 4-20mA current loop for process control. The primary use of the vision system is in classifying particles by size but can also be used to obtain shape parameters. Typical size information for polymeric bead samples is presented to demonstrate the system's capabilities. The flow cell vision system has numerous advantages over traditional size measurement techniques including speed, resolution, and ease of replication.

INTRODUCTION

Polymeric beads are used in ion exchange resins(1), combinatorial chemistry(2), and can be molded to form many common plastic goods(3,4). Often the bead size is an important consideration in functionality of the finished product. Therefore, polymeric bead manufacturers wish to control and characterize the beads they produce(5). For example, when beads are to be used in an ion exchange resin, the use of smaller beads results in a cost savings due to a large surface area to volume ratio. The beads must however be of some minimum size so that they do not to flow out of the ion exchange column. The user of these beads therefore desires them to have a tight size distribution about a specific size. The manufacturer who can provide this more efficiently, either by controlling his process directly or providing a tightly controlled quality assurance to his customer, obtains a competitive advantage over other producers.

Traditional particle sizing methods involve taking a sample from the line to the lab, analyzing it, and reporting the results back to the plant where adjustments can then be made to control process output. This can sometimes result in delays of anywhere from a few minutes to several hours. A need to dry the product before analysis can also add to the lag time between when the beads are made and when their characteristics are known. The vision based in-line measurements described here present an alternative form of analysis that can significantly cut the lag time involved with many traditional analysis schemes. These measurements utilize a sensor rated for the process environment that is capable of imaging optically difficult materials and an on site or remotely located computer based Vision Processor. The processor makes measurements that allow real time control of the process. The system includes components for driving a traditional 4-20 mA analog control loop for in-line process management. It is also designed and equipped to share all measurement data with other users on an existing computer network.

An additional advantage of the vision based size measurement described here is that the measurements are not restricted to a few known sizes. When a traditional sieve measurement is made, particle size distribution is known only at the sizes of the screens used to make the measurement. The vision system allows for particle size measurement in between the screens giving a great deal more information to the user. This is particularly beneficial when the size distribution of a sample is very tight and falls almost entirely between two screens or when the tolerance of the screen is greater than the differences in particle sizes of the distribution.

EXPERIMENTAL

The system used in this investigation was an integrated flow cell that allowed a slurry of particles to flow past the imaging sensor for real time measurement (Figure 1). The key elements of this system are the CCD camera, functioning as the sensor, and the computer based vision processor that extracts measurement data from the process images provided by the camera and provides output data to the operator. An appropriate lighting system is essential for obtaining accurate results and should be integrated into the flow cell configuration along with the camera. While the camera, flow cell and lighting can be exposed to the production area, the computer should be located in a general-purpose or control room away from the process environment.

The imaging sensor used was a black and white process camera with 1/100,000 s^{-1} shutter speed and all auto gain control functions disabled. A microscope zoom lens system was used to obtain images of the beads that were suitable for image processing. The flow cell gap between imaging surfaces was set to 500 microns in order to allow the largest particles to flow through the cell without plugging. A larger gap could be selected if product characteristics warranted.   The computer measures in terms of pixels. A pixel scale factor in terms of a common measurement unit needs to be set in order to make measurements in that desired unit. The flow cell system was calibrated for measurement in microns by using a visually verifiable image of a calibration scale to determine the size of the pixel. A pixel scale factor of 2.546 microns per pixel was used for all measurements made. Product images shown here present a back lighted 1600 x 1200 micron field of view of the particles as they flow through the cell.

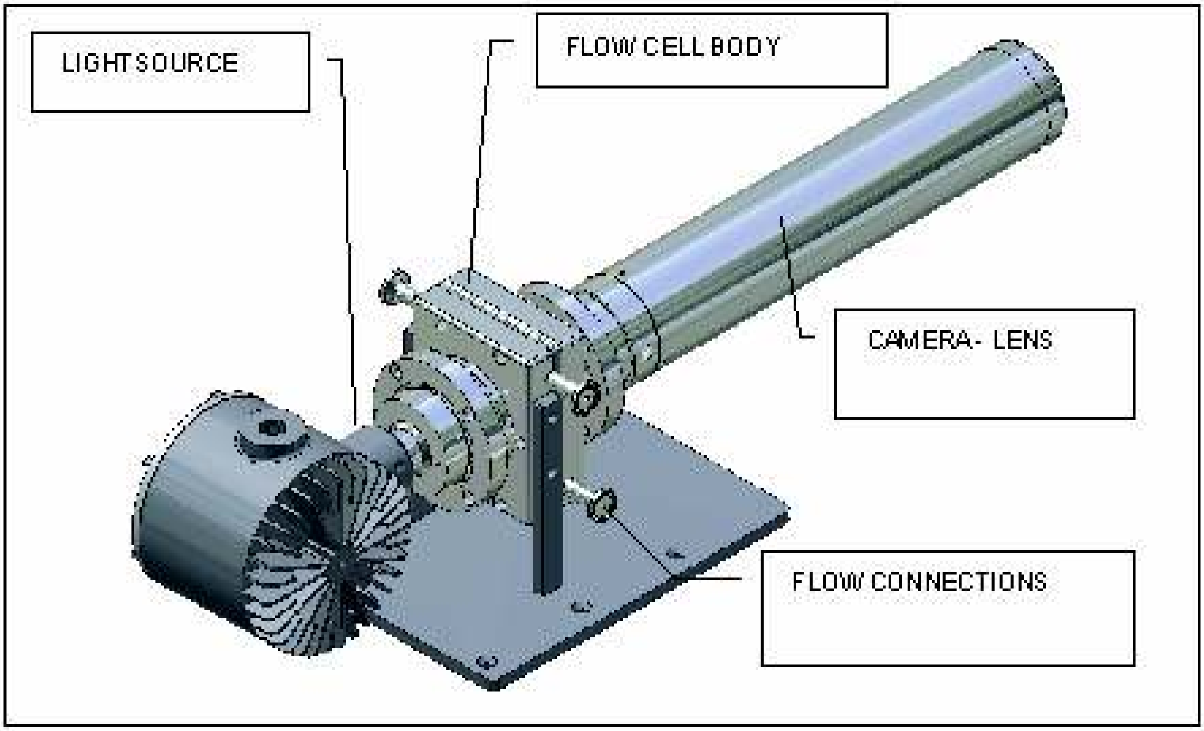

FIGURE 1 - INTEGRATED FLOW CELL FOR SLURRY ANALYSIS.

While polymer beads are usually grown in solution, the beads analyzed were obtained in a dried form. The polymer beads were therefore diluted to an approximate concentration of 2.5 grams of beads per liter of water to allow introduction into the flow cell. This concentration gave sufficient particle separation to allow for single particle identification and measurement. In order to simulate an online system, the samples were cycled through the cell until a statistically significant number of particles were imaged.

RESULTS

A key benefit of the vision-based measurements detailed here is the ability to visually verify the proper operation of the camera/light as well as the vision processing computer. The computer display monitor can provide a process image with measurement indicator overlaying the camera image. This allows operators to view actual process conditions in the event than any unexpected results are presented. Figure 2 shows a process image of the aggregate slurry as it passes through the flow cell. Figure 3 presents the digitized image of the slurry and Table I lists particle measurement dimensions for the particles in the image.

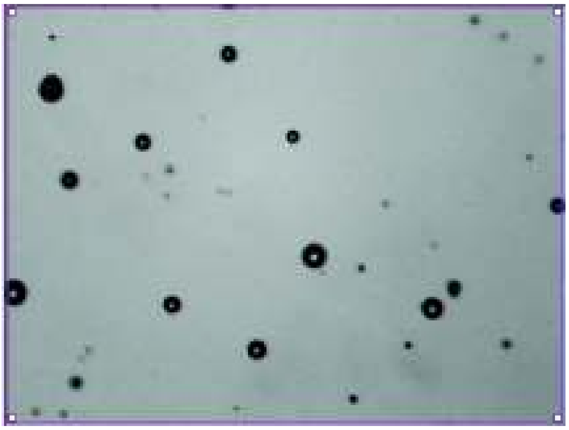

FIGURE 2 - IMAGE OF PARTICLES IN SOLUTION.

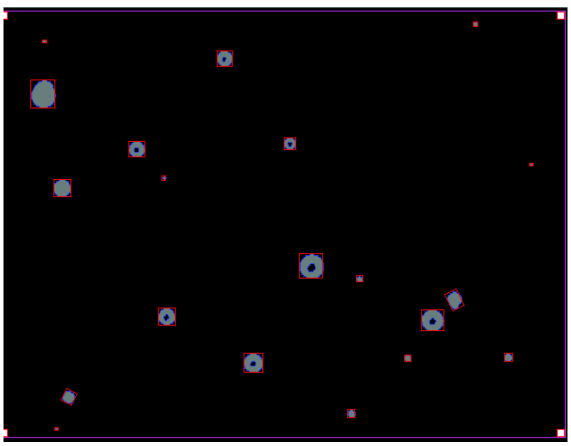

FIGURE 3 - DIGITIZED IMAGE OF PARTICLES IN SOLUTION.

TABLE I - PARTICLE DIMENSIONS.

	Area (micron squared)	Perimeter (micron)	Major Axis (micron)	Minor Axis (micron)
1	220.4	48.37	15.28	15.28
2	142.6	38.19	15.28	10.18
3	1672	178.2	45.83	45.83
4	4421	236.8	81.47	71.29
5	985.3	147.7	35.64	35.64
6	1672	188.4	48.37	45.83
7	103.7	30.55	12.73	10.18
8	129.6	35.64	15.28	12.73
9	2100	160.4	50.92	50.92
10	3630	292.8	71.29	68.74
11	311.1	61.1	20.37	20.37
12	1569	140	51.6	42.51
13	3060	254.6	66.2	61.1
14	1906	203.7	50.92	50.92
15	376	63.65	20.37	20.37
16	531.5	78.93	25.46	25.46
17	2398	221.5	56.01	56.01
18	1011	109.5	37.87	35.64
19	479.7	73.83	25.46	22.91
20	103.7	30.55	12.73	10.18

After visual verification of a few images to verify measurement data for the slurry of beads, the sample was run through the cell to obtain a set of images from which continuous particle size distribution measurements could be made. Figure 4 presents the particle size distribution for a set of the beads with an average size of 150 microns while Figure 5 presents the particle size distribution for a smaller sample of beads. The plots give a continuous particle size distribution in terms of the volume percentage of particles with a minor axis dimension less than the size specified on the x-axis of the plot. This distribution was plotted for over 3,000 particles in the bead samples. The error bars shown with the sieve data in the charts represent a +/- 3% uncertainty in the size of the screen openings. This uncertainty is consistent with the error allowed by the American Society for Testing and Materials standard E-11 (Standard Specification for Wire Cloth and Sieves for Testing Purposes) and the International Standards Organization standards that sieves are manufactured to(6). The vision system avoids this type of replication uncertainty by calibrating to a known standard when determining the pixel scale factor.

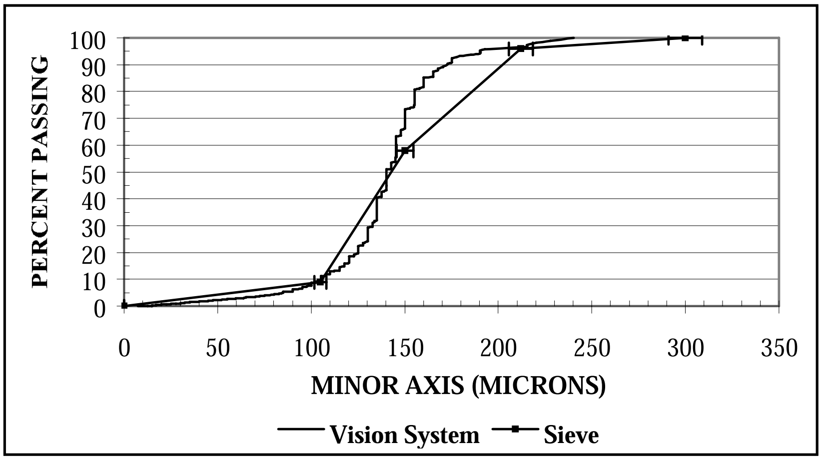

FIGURE 4 - PARTICLE SIZE DISTRIBUTION FOR SLURRY #1.

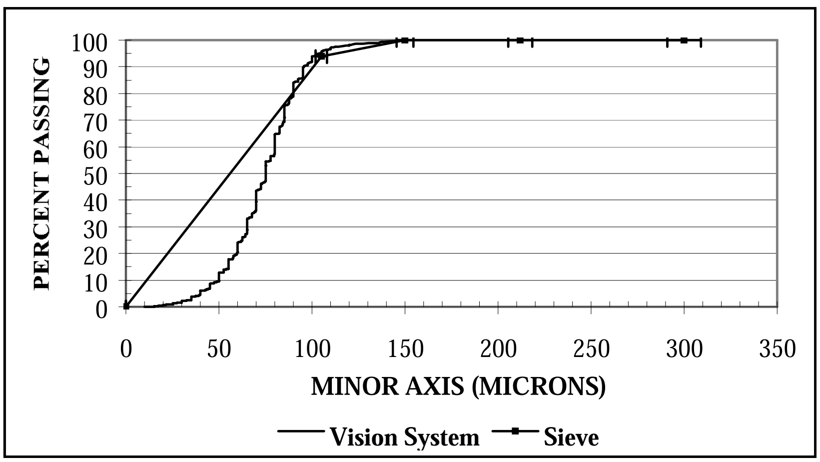

FIGURE 5 - PARTICLE SIZE DISTRIBUTION FOR SLURRY #2.

It is interesting to note that in both figures the sizes do pass through the error bars of the screens except for the 300 micron sieve in Figure 4. This occurs because the entire product is actually smaller than 225 microns and therefore the plot should not be expected to intersect in the 300 micron size range. Also noteworthy is that the assumption of a straight line function between the sieve data points may not be realistic in that there is no knowledge of the particle sizes in these regions of the plots. This leads to seeming discrepancies between the two data measurement techniques in that some areas of the distributions, like the particles less than 100 microns in Figure 5, do not appear to match. This occurs because the sieves used do not have the resolution to make measurements in this area. Figure 6 gives an extreme example of this where a very tight distribution of beads is sized. In this plot, the screen data shows only that the entire sample of beads are somewhere between 210 and 300 microns while the vision system shows that the true size distribution is much tighter than this and centered not on the midpoint of the two screens but at approximately 235 microns.

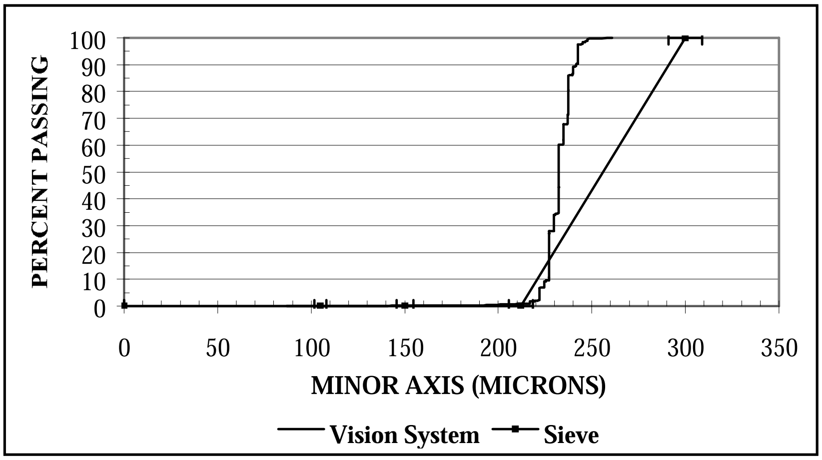

FIGURE 6 - PARTICLE SIZE DISTRIBUTION FOR SLURRY #3.

Table II presents summary data for three trials using the samples in the first two distribution curves shown above. This table lists the particle minor axis dimensions in microns for the 10, 50 and 90 percent passing points in the particle size distributions. The data in the table shows that in general the sizes at a given percent passing are tightly distributed for the 10 and 50 percent passing points and slightly more dispersed at the 90 percent passing point in the larger of the two distributions. Summary statistics like these points are used to design a laboratory or on-line method of data analysis that can be used to control the bead growth process in the plant. This will allow for greater control toward optimization of bead sizes in the production environment.

TABLE II - SUMMARY DATA IN MICRONS FOR DISTRIBUTION CURVES.

Percent Passing		10%	50%	90%
Slurry #1	Avg.	103.8	138.0	183.6
	σ	2.4	2.2	8.6
Slurry #2	Avg.	50.2	76.3	96.8
	σ	0.5	0.0	0.4

The vision based system has an additional advantage in that it can provide shape information in addition to size information. In the case of beads, a spherical shape is desired so the aspect ratio, defined as the major axis divided by the minor axis, should be close to unity in good product. Inspection of the data in Table I shows that this is the case for most of the particles. Particle 4 however has an aspect ratio of 1.14. This particle is located in the upper left hand corner of Figure 2. It can be seen that this large particle is certainly more oblong than the other particles in the image. Measurements similar to this can be made on large numbers of beads at the same time size measurements are made. The fraction of particles with a desired shape will then allow for enhanced product quality control.

DISCUSSION

The primary control signal output from the vision processor is the 4-20 mA analog output that interfaces with the process controller. This is a well-established industry standard interface. This output mechanism allows for automated online control of the process environment to be achieved using the vision system. As the flow cell is designed to work with liquid slurries, it can be attached to the process vessel where the beads are grown, remove a sample of beads, measure them and then return the beads to the process vessel. This can be incorporated as a continuous loop to provide process control measurement data and a signal can be sent via the 4-20 mA to trigger process changes. Alternatively, beads can be sampled and then taken to an off line flow cell for measurement and the data from this measurement can then be used for process control or quality assurance. Even in this configuration, a time advantage over traditional sieve measurement techniques will be realized.

In certain situations, such as at system startup or when undesirable size or shape readings occur, the operator may wish to look at a single snapshot of the beads as they flows through the cell. For this purpose, additional readouts of the vision system's measurements are available in the form of graphical displays. This permits the operator to visually confirm numerical data with the process images. Where a record of numerical data is needed for archive, the computer files are stored on the hard drive can be managed by network connection to the facility archive server. Where custom user calculations are required to calculate a final control value from the raw measurement data, a spreadsheet link to the measurement data is made and the spreadsheet then sends the calculated result as a 4-20 control value.

CONCLUSION

The vision based 2-D Particle analysis approach described here offers a data acquisition technique that permits real time process control in polymeric bead growth operations. The system's ability to continuously monitor the process can help to achieve efficient control of the production unit so that optimal product characteristics are obtained. The vision system can monitor particle sizes with resolutions that would be difficult and costly to perform with traditional sieve measurement techniques and avoids uncertainties related to allowable variance in the screen manufacturing process. It can also be used to identify shape factors in polymeric beads and affords a time savings to the user because measurements are made on wet slurries of beads and drying is not necessary. These factors will lead to cost advantages through the efficient the production of beads with the correct characteristics for their intended use.

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