CZECH TECHNICAL UNIVERSITY IN PRAGUE

FACULTY OF MECHANICAL ENGINEERING
DEPARTMENT OF PROCESS ENGINEERING

MEASURMENT OF PARTICLE SIZE IN INDUSTRIAL SUSPENSIONS

BACHELOR THESIS

2016 LUCIE MARCALÍKOVÁ

I declare that I have developed the bachelor thesis independently under the leadership of supervisor of bachelor's thesis, and I made references to all of the used documents and literature.

In Prague on ……………… …………………

Lucie Marcalíková

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ANNOTATION

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Annotation in Czech: Práce představuje různá technická řešení měření velikosti, počtu částic a koncentrace v průmyslových suspenzích. V závěrečné části se nachází návrh měření v reálném procesu a v laboratorním experimentu.

Annotation in English: This paper presents various of technical solutions measurement of size, number and concentration of particles in industrial suspensions. The final part is possible setup for measurements in real process and in laboratory experiment.

Key words: particle mesuring, particle counter, online analysis, optical particle measuring

Utilization:

CONTENT

1. INTRODUCTION

Particles, crystals, and droplets occur in many chemical processes, across a range of industries, and often pose challenges for scientists and engineers who are tasked with optimizing product quality and process efficiency. Characterizing particle properties effectively, in particular particle size and shape and count, allows processing problems to be solved and product quality to be improved. Historically, scientists have relied on off-line particle size analysers, such as laser diffraction or sieving to perform this type of characterization. During past years, newer technologies have emerged, which describe particle size and count in real time, as particles naturally exist in process with higher measuring quality, in less time at a lower total cost. In process measurement of particles can reduce the error associated with offline sampling, and can provide continuous information about how particles behave under changing process conditions, allowing scientists to understand and optimize difficult processes using evidence-based methods.

This paper will introduce some of the most common online methods and devices in process particle measurement with special focus on solid particles in liquid medium. The determination of optimized process parameters is critical to ensure the correct particle size and count that can be obtained consistently.

1.1. Specification of medium

This paper has special focus on two types of medium. First is quench oil containing coke particles with process temperature 160°C and second is quench water containing coke particles and tar drops with process temperature 80°C. Other parameters like pressure, flow rate, etc. are same for oil and water and they are described in table [1] below. Particle concentration is in ppm scale and expected size is 100 – 2000μm.

1.2. Critical Restrictions

While choosing convenient device or method, there are some that must be taken into consideration.

1.2.1. Particle Distribution Accuracy

When considering the purchase of a particle counting or monitoring device, one must decide if particle counting or trending of the concentration of particulate is the ultimate objective. A particle counter may be required if the particle count will be used to certify fluid cleanliness or if general research is the intent.

Optical particle counters estimate the size of each particle based upon the change in light energy across the sample flow area in the instrument. A large energy depression, if it is a light blockage particle counter, or a large energy spike if it is a light scattering particle counter, suggests a proportionally large particle. While error can result due to the orientation of the particle and other factors, the optical method is generally effective at estimating the size of each particle. The pore blockage counter does not count particles individually; rather it measures the collective influence the entire population of particles has on the fluid flow through the screen. A particle count requires a measure of several different sizes of particles. The optical method may require considerably more attention to procedural detail and may not be suitable for some fluids, but it does offer the flexibility to analyse the particle size distribution.

Another restriction is particle concentration itself. Some methods are made for measuring in ppm scale, some requires bigger volume of particles, usually 10-70%, to have reliable data.

1.2.2. Fluid Types Tested

In addition to the need for distribution accuracy, one must also consider the fluids used and typical contaminants in the various applications to be monitored. Optical particle counting is influenced by a number of factors - aeration, water, dark fluids, heavily contaminated fluids and sample preparation. Some optical methods have limit of fluid transparency and without powerful light source it is impossible to measure dark oils.

1.2.3. Procedural Requirements

When using either light or mesh technology units online, it is necessary to allow sufficient flushing time to ensure the unit’s sampling hoses are thoroughly clear before accepting a reading. The flush time on some units is fixed, while on others, it can be overridden by the user, which can be advantageous when doing research at a fixed location. When using an optical particle counter online, ensure that the line pressure is sufficient to eliminate interference from air bubbles, or that the particle counter itself deals with the air.

1.2.4. Application And Use

The choice for an onsite machine condition-monitoring application is between bottle samples and direct online measurement. For walk-around measurements with a portable unit, primary and secondary connections to a system allow nonconforming tests to be repeated immediately and possibly troubleshoot the problem (such as inspecting filters and breathers). This will require quick-connect fittings, and some units may require a custom manifold. [1]

2. POSSIBLE METHODS

In this chapter there will be introduced four most common online particles measuring methods.

2.1. Industrial computed tomography

2.1.1. Principle

Industrial computed tomography (CT) scanning is any computer-aided tomographic process that uses irradiation (usually with x-rays) to produce three-dimensional representations of the scanned object both externally and internally. In principal CT is the most effective available method for 3D-analysis of any object as long as there are differences in the density and/or atomic number of the components to inspect.

Industrial CT scanning has been used in many areas of industry for internal inspection of components. Some of the key uses for CT scanning have been flaw detection, failure analysis, metrology, assembly analysis and reverse engineering applications.

There are two types of scanners, fan/line beam and cone beam.

1. *Fan/line beam scanners-translate*

Line scanners are the first generation of industrial CT Scanners. X-rays are produced and the beam is collimated to create a line. The X-ray line beam is then translated across the part and data is collected by the detector. The data is then reconstructed to create a 3-D volume rendering of the part.

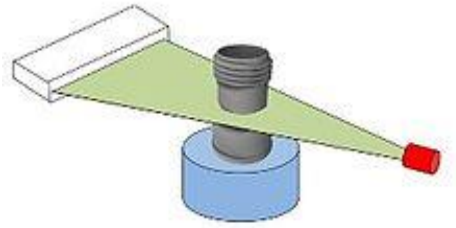


Figure 1: Line scanner [2]

2. *Cone beam scanners-rotate*

During the CT scan the part is placed on a rotary table. As the part rotates the cone of X-rays produce about 1300 2D images which are collected by the detector. The 2D images are then processed to create a 3D volume rendering of the external and internal geometries of the part. [2]

Industrial computed tomography allows for a comparison of parts to one another or parts to CAD data. The deviations for both external and internal geometries can be shown on the surface colour map chromatically on the 3D representation or by whisker plots in the 2D windows. This process is beneficial when comparing the same part from various suppliers, studying the differences in parts from one cavity to another cavity from the same mould, or verifying the design to the part. All colour coordinated particles within casting are voids/ porosity/ air pockets, which can additionally be measured and are colour coordinated according to size. [3]

2.1.2. Real Process solution - Tracerco

Figure 2: Tracerco subsea technology [4]

Tracerco company released two products, to deliver high resolution CT scans of subsea pipelines. The technology works without the need to remove pipe coating materials and can both accurately locate blockages and in- line analyse its precise nature.

This device is measuring the wall thickness of pipelines which can be made in minutes with a high resolution image of pipeline contents being provided from an external scan of the pipeline. [4]

2.1.3. Conclusion

CT is fully developed for laboratory applications but not yet satisfactorily for field applications. The reasons for restrictions in mobile usage are the need of full access from all directions to the object and the requirement of several hundred projections, taken at different angles through the object. Access and measurement time are restricted in most cases.

Technology is suitable for gas or liquid filled pipes and multiphase flow does not affect the results. However measuring resolution is to 2 mm, which is not enough for our application.

2.2. Spectroscopic Measurement

2.2.1. Principle

The principle to measure the properties of the matter is to observe the structure of the molecular. The interactions of various types of electromagnetic waves with matter provide the information of the molecular bands and movement behaviours. The electromagnetic wave in different range of wavelengths from gamma rays to radio waves can be used to excite the molecular. It can be collected amount of absorbance and reflectance signals to estimate the structures of samples. Each range of wavelengths or frequencies reacts with matter in a different way.

In the spectroscopic system the incident light beam targets to the samples required to be analysed. The response light or energy loss in other words, can be collected in very different ways, such as reflection, transmission and scattering light. The practical decision to use which parts of response strongly relies on the purpose of analyser and the sample characteristics. Two most common kinds of setup are transmittance and reflectance. For example, the transmittance setup is usually used for transparent and clean liquid samples and the reflectance setup for dark and solid samples. [4]

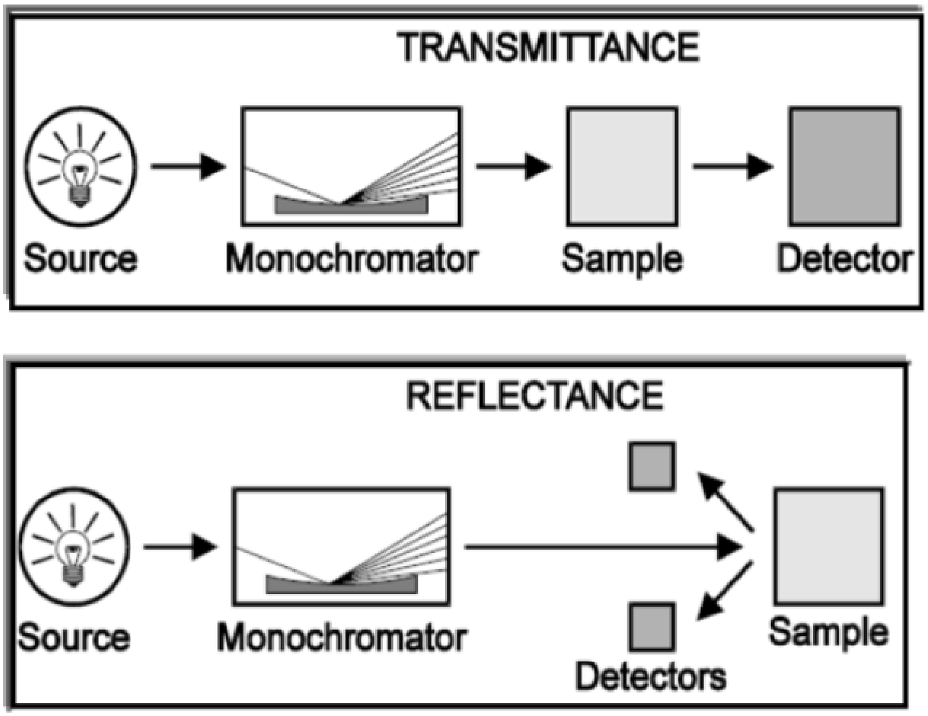


Figure 3: Two most common kinds of setup

2.2.2. Obstacles In Real Process

Probably the biggest problem is that this application is still in the experimental stage for more reliable results. On the other hand it is perspective technology which might have success in near future.

The major difficulties are caused by inconsistent material /heterogeneous flow, high viscosity, probe erosion and low concentrations of others particles which need to be measured accurately. The instruments must be operated under a vacuum which is not always practical for industrial applications. Usually chemometric software packages and a set of known samples with primary data are required to build multivariate statistical models in order to identify the unknown samples.

2.3. Pore Blockage

2.3.1. Principle

Pore blockage particle counters are used as on-site particle counters for in-service machinery oils. They employ a fine mesh whereby particulate accumulates on the mesh. In this method, a volume of fluid is passed through a mesh screen with a clearly defined pore size, commonly 10 microns. These particle counters are based upon either a constant flow or constant pressure design.

One instrument measures the flow decay across the membrane as it becomes plugged while pressure is held constant, first with particles greater than 10 microns, and later by smaller particles as the larger particles plug the screen. The second measures the rise in differential pressure across the screen while the flow rate is held constant as it becomes plugged with particles. In both cases, the particle count distribution is estimated by extrapolation and both instruments are tied to a software algorithm, which turns the time-dependent flow decay or pressure rise into an ISO cleanliness rating. [1]

2.3.2. Obstacles In Real Process

Pore blockage particle counters are rarely used by commercial laboratories due to the limited data generated but can be of great use where interference from water, soot or additives is highly prevalent in the samples. Disadvantage is that it only measures a single size channel and distribution dependent at the pore size being measured.

While pore block particle counters do not suffer the same problems as optical particle counters with respect to false positive caused by air, water, dark fluid, etc., they do not have the same dynamic range as an optical particle counter, and because the particle size distribution is roughly estimated, are dependent on the accuracy of the algorithm to accurately report ISO fluid cleanliness codes. Nevertheless, they accurately report the aggregate concentration of particulates in the oil, and in certain situations, particularly dark fluids such as diesel engine oils and other heavily contaminated oils, pore block particle counting does offer advantages. [6]

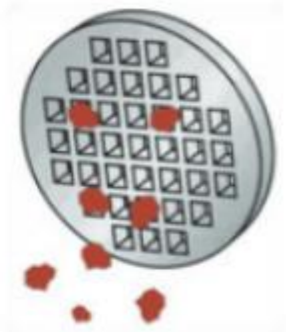


Figure 4: Pore block counter [6]

2.4. Optical Measurement

2.4.1. Principle

Optical particle counting is based upon either light scattering, light obscuration, or direct imaging. A high intensity light source illuminates particles as they pass through the detection chamber and the light source (usually a laser or halogen light) and if light scattering is used, then the redirected light create a shadow and is detected by a photo detector. The drop in voltage produced by the photocell is directly proportional to the size of the shadow and hence the size of the particle passing through.

In case of direct imaging a halogen light illuminates particles from the back within a cell while and high definition and magnification camera records passing particles. Recorded video is then analysed by computer software to measure particle attributes. If light blocking (obscuration) is used the loss of light is detected. The amplitude of the light scattered or light blocked is measured and the particle is counted. Direct imaging particle counting employs the use of a high resolution camera and a light to detect particles.

Vision based particle sizing units obtain two dimensional images that are analysed by computer software to obtain particle size measurement in both the laboratory and online. Along with particle size, colour and shape analysis can also be determined.

Laser optical particle counters are generally considered to be slightly more accurate and sensitive than white light instruments. [2]

2.4.2. Detection Methods

There are several methods used for detecting and measuring particle size or size distribution — light blocking (obscuration), light scattering, direct imaging and pore blockage.

2.4.2.1. *Light Blockage Particle Counter*

The light blocking optical particle counter method is typical useful for detecting and sizing particles greater than 1 micrometre in size and is based upon the amount of light a particle blocks when passing through the detection area of the particle counter. This type of technique allows high resolution and reliable measurement.

Laser light blocking particle counters are the traditional instruments used for in-service oil analysis. A light source, typically a laser, passes through a sample. The light is partially blocked by particles so less light reaches the photodetector array, resulting in a change in voltage proportional to the area of the particles. The photo detector technology is the same principle used in garage door openers and it is easy to automate. The photo detector results contain measurement errors caused by the presence of water and air bubbles within the oil sample. [7]

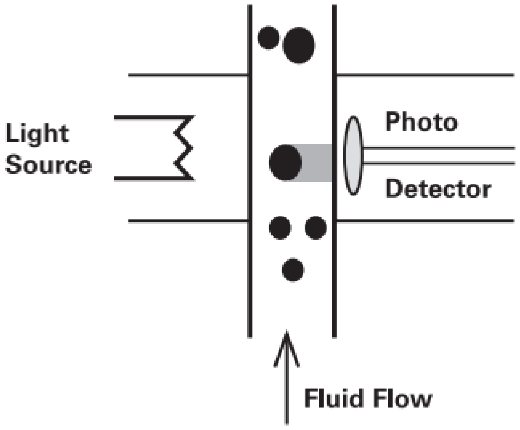
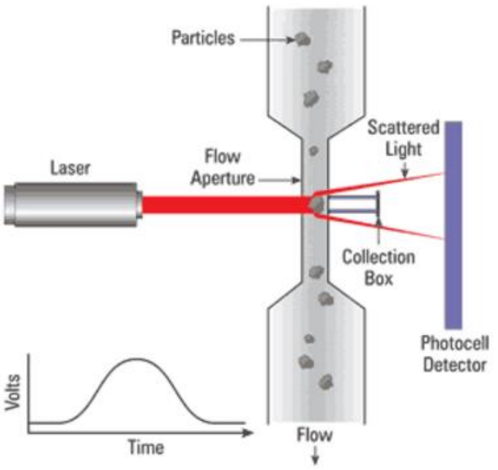


Figure 5: The light blocking optical particle counter [7]

2.4.2.2. *Light Scattering Method*

The light scattering method is capable of detecting smaller sizing particles. This technique is based upon the amount of light that is redirected by a particle passing through the detection area of the particle counter. This redirection is called light scattering. Typical detection sensitivity of the light scattering method is 0.05 micrometre or larger.

In a laser-based instrument, due to the near-parallel nature of the laser beam, light scattering from the unimpeded laser beam is minimal because it is focused into a beam stop - until a particle passes through the instrument. As the laser strikes the particle, light scatters and hits the photocell. Just like a white light instrument, the change in voltage across the photocell is directly related to the size of the particle. [1]



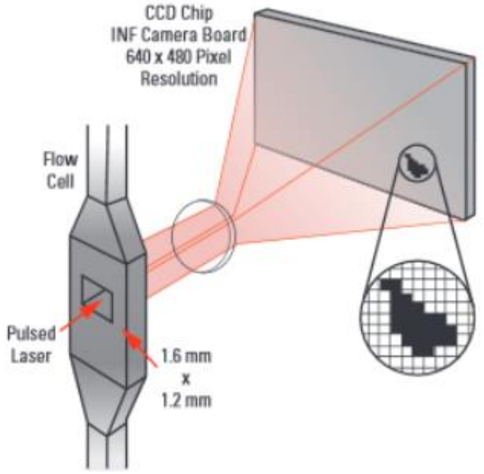


Figure 6: The light scattering method [1]

Figure 7: Direct Imaging Particle Counter [1]

2.4.2.3. Direct Imaging Particle Counter

Direct imaging is a technique that uses the light emitted by a laser as a source to illuminate a cell where particle are passing through. The laser illuminates the sample, and an optical lens magnifies the laser light. The technique does not measures the light blocked by the particles but rather measures the area of the particles functioning like an automated microscope. A pulsed laser diode freezes the particle motion and CCD video camera captures the images of the sample and stores them in memory. These images are analysed for size and shape. An equivalent circular diameter or ECD is calculated for each image and particle count and size distribution is reported along with ISO codes. The capability of direct imaging systems to capture the actual wear particle silhouette allows for an ‘Automated Ferrography’ capability for wear particle classification. All particles larger than 20 µ are classified by a neural network in the categories of cutting, fatigue, severe sliding, non-metallic, free water and fibres. Identifying the type of wear particle and providing particle count, size distribution and severity of each of the abnormal wear mechanisms complements information provided by other instrumentation technologies such as ferrous monitoring and analytical ferrography. Imaging systems can distinguish between solid particles, water droplets and air bubbles in oil for all particles greater than 1 µ. Water and air bubble counts are subtracted from the measured particle count to yield a true net particle count. No calibration of system is required (intrinsically correct). [1]

3. POSSIBLE DEVICES

After searching for a possible solution for our case, only optical methods give satisfactory results. There are many companies providing optical solution for measuring particles with different systems. I choose three of them, most suitable for our application. All of them have different scanning method and different limitations, but most of the parameters fits to our measurement requirements such as ability to measure in hazardous environments (ATEX) and full in-line measuring at full process concentrations and does not require sampling. Characteristics of each device can be found in the attachment. [1]

3.1. Probe system (Mettler Toledo)

Probe-based instrument using focused beam reflectance measurement technology is insert into a process stream for direct measurement of particles as they naturally exist in the process. Probes can be applied across a range of scales and measurement is taken every few seconds, allowing discrete distributions. Statistics from each distribution can then be trended over time allowing scientists to monitor process trajectory in real time.

3.1.1. Principle

The probe is inserted directly into process streams, at an angle, to ensure particles can flow easily across the probe window where the measurement takes place. A laser beam is launched down the probe tube through a set of optics and focused to a tight beam spot at the sapphire window. The optics rotate at a fixed speed resulting in the beam spot rapidly scanning across particles as they flow past the window. As the focused beam scans across the particle system, individual particles or particle structures will backscatter the laser light to the detector. These distinct pulses of backscattered light are detected, counted, and the duration of each pulse is multiplied by the scan speed to calculate the distance across each particle. This distance is defined as the chord length, a fundamental measurement of the particle related to the particle size. Typically thousands of particles are counted and measured per second, allowing a precise and highly sensitive chord length distribution to be reported in real time. The chord length distribution tracks how particle size and count change from the beginning, until the end of a process. Statistics from each chord length distribution, such as counts in fine and coarse size classes, can be trended over time.[8] [9]

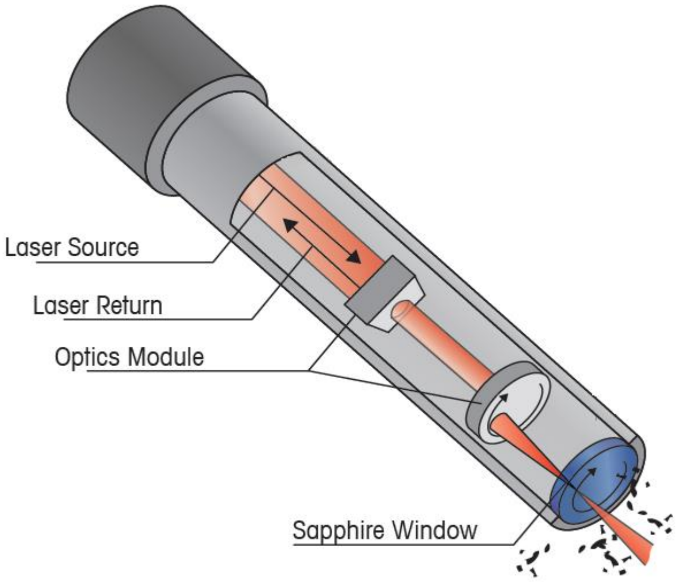


Figure 8: Probe: -based instrument [10]

3.1.2. Main restrictions

The main problem (for our use) with this method is particle concentration. Typically the particle concentration has to be more than 10% to have reliable data. Our concentration is in ppm range and output curve would be very inaccurate and changing in time.

3.2. Laser beam (Malvern)

Insitec systems use the technique of laser diffraction to rapidly measure particle size directly in the process stream. When applied to wet systems, complete particle size distributions can be measured in less than one second, giving instantaneous monitoring. Even processes with fast dynamics can be tracked effectively.

3.2.1. Principle

Using the established principle of spatial filter velocimetry, size and velocity can be simultaneously extracted from particles as they pass through a laser beam, casting shadows onto a linear array of optical fibres.

A burst signal is generated due to the particle crossing fiber bundles labelled “burst a” and “burst b”. The frequency of this signal is measured by photodetectors and is proportional to the particle velocity v. Knowing the spatial filter constant g, the velocity v can be calculated. As the particle passes through the beam, a secondary “pulse” signal is generated by a single optical fiber. Knowing the time t of the pulse signal, and the velocity v of the moving particle, the chord length x of the particle can be calculated.

Scattering algorithms mathematically correct for the concentration-dependent effect of the source light interacting with not one, but several, particles before reaching the detectors. More samples can be measured without dilution; concentrated solutions require less pre-treatment. [11] [12] [13]

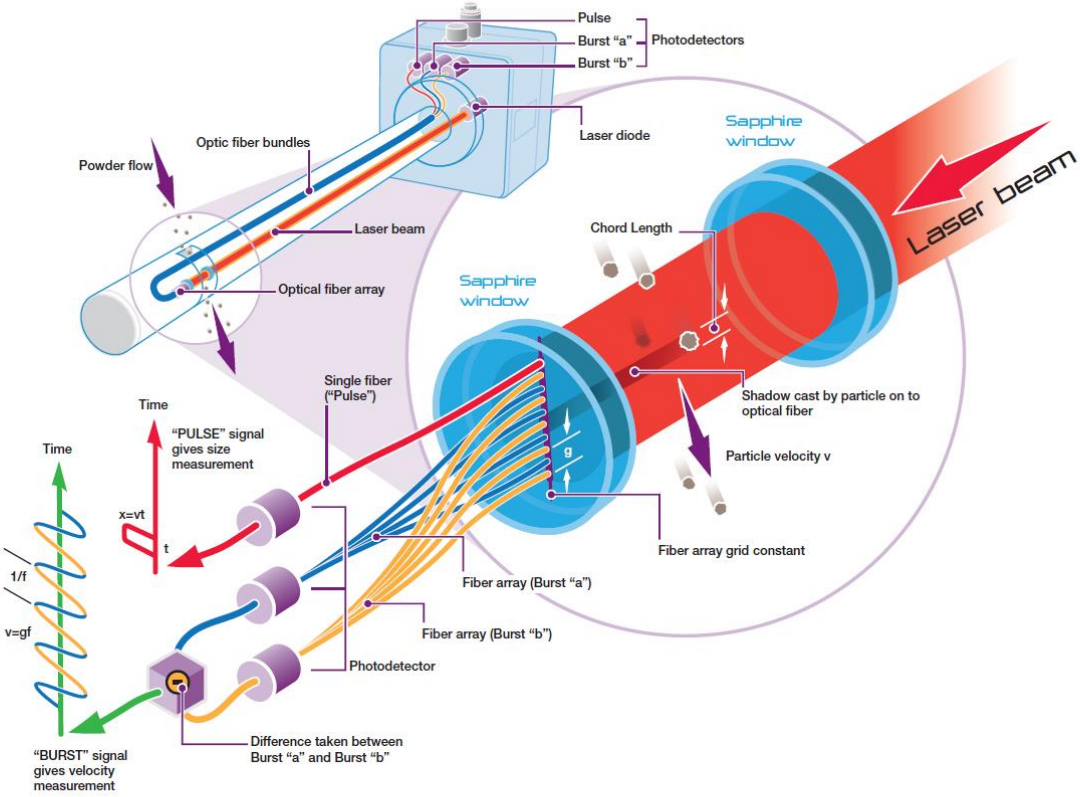


Figure 9: Laser diffraction [13]

3.2.2. Main restrictions

This method would be more suitable for measuring quench water, because laser beam needs certain transparency of medium to be able catch the signal. However medium with quench water is with coke particles as well as with tar drops and system is not able to recognise the difference between them. Another problem is again the particle concentration, which is the same as with the case before. This device has faster scanning system, but concentration still has to be in in percentage to get reliable data.

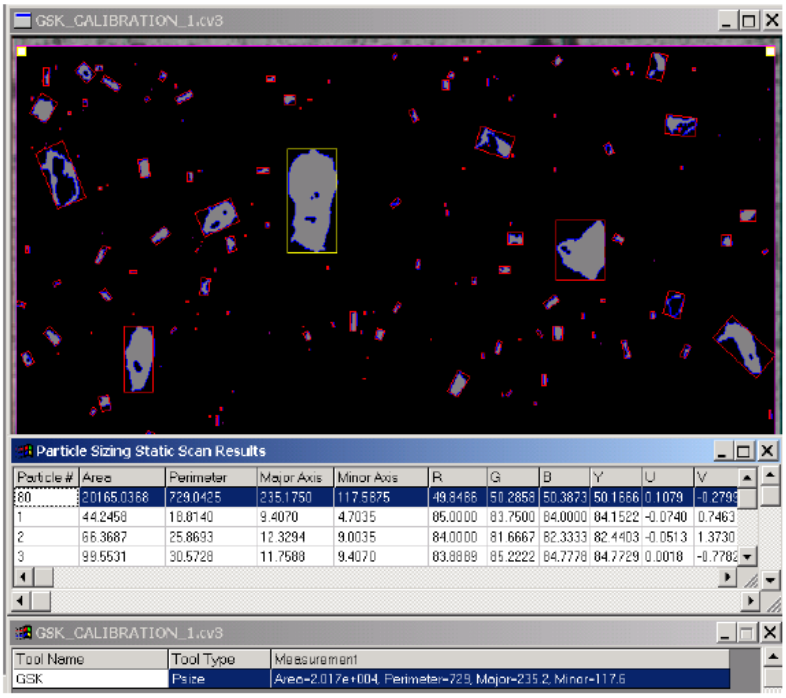
3.3. Light + CCD camera (JM Canty)

All particles have shape and it is impossible for a single mode instrument, such as a laser, to measure the size of different particles. This method allows the operator to see the process and make intelligent selections of how best to gather accurate data. A Canty Vision System Camera and Light remotely monitor product under actual process conditions. Both microscopic camera and high intensity light source are sealed from process behind a hermetic fused glass seal and the resulting real time video is digitally transmitted and analysed using software, where the suspended particulate (water, oil, solids, gas bubbles etc.) is measured under a number of different parameters to provide size, shape and concentration data.

3.3.1. Principle

Particles are sent through the flow cell body, back-lit with a high output light and particle images are collected in real time by the CCD camera. After the image is digitally transmitted to a PC with software for analysis. The image is then broken down into individual pixels. The colour difference between the particles and the background allows software to determine the parameter of the particle, as well as the major axis, minor axis, area, and other characteristics about the particles dimensions.

Once the software determines the particles size and shape, the software can perform further analysis on the individual particles. The analysis includes particle filters to enable users to determine when particles are dissimilar or nonconforming to the entire distribution of particles. [14]



Particle #	Area	Perimeter	Major Axis	Minor Axis	R	G	B	Y	U	V
80	20165.0368	729.0425	235.1750	117.5875	49.8486	50.2858	50.3873	50.1666	0.1079	-0.279
1	44.2458	18.8140	9.4070	4.7035	85.0000	83.7500	84.0000	84.1522	-0.0740	0.7463
2	66.3687	25.8693	12.3294	9.0035	84.0000	81.6667	82.3333	82.4403	-0.0513	1.3730
3	99.5531	30.5728	11.7588	9.4070	83.8889	85.2222	84.7778	84.7729	0.0018	-0.778

Tool Name	Tool Type	Measurement
GSK	Psize	Area=2.017e+004, Perimeter=729, Major=235.2, Minor=117.6

Figure 10: Particle sizing [14]

3.3.2. Design

Both designs works on the same principle, only difference is process connection and measured volume. Flanged system measuring whole volume of the pipe, whereas portable system creates bypass.

3.3.2.1. Flanged system

This centre pipeline measurement is most representative, because it allows to scan the whole volume in pipe. System uses variable insertion measurement gap, which allows the optical fused pieces to be located in the centre of the fluid stream.[15]

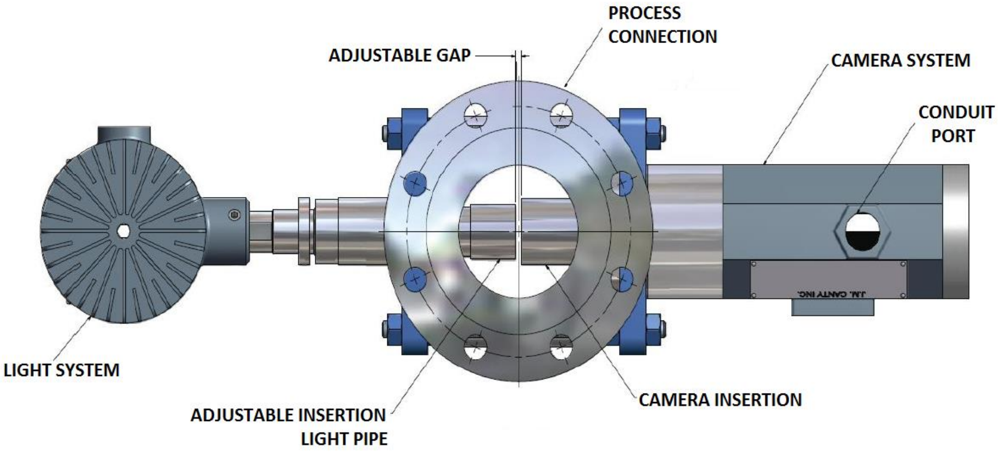


Figure 11: Flanged system [15]

3.3.2.2. Portable system - fast loop bypass

When using portable system, it is insured that the sampling point is taken from a vertical main line with upward flow. As from the sampling guidelines from DECC (regulatory body in the UK), sampling should be taken from the centre third of the pipeline. To ensure this a sampling probe (“L” shaped) should be inserted into the centre third of the pipe with the probe facing upstream. This ensures a representative sampling line form mainline. [16] [17]

Figure 12: Fast loop bypass [16]

3.3.3. Main restrictions

There is no information so far what is happening inside the pipe and if the medium is homogeneous and well mixed so the bypass sampling from one level of the pipe doesn’t have to be representative. Flanged system is scanning whole volume of the pipe which provides also data from different levels in pipe in time.

4. MEASURING IN REAL PROCESS

For measuring our case the best option seems to be using Canty flanged system, specifically “Oil in water, water in oil, multiphase oil, produced water, tar sands measurement systems” (attachment [2]). Model VM6CA11BF3V fits to measuring point „3&4“ on pipe DN300. This device is fulfilling all requirement for both medium and particle measuring requirements (refer to the attachment [1][2]). In order to have optimal flow rate, pipe diameter in measuring point might be modified if necessary. Device is also designed for measuring in hazardous environments (ATEX).

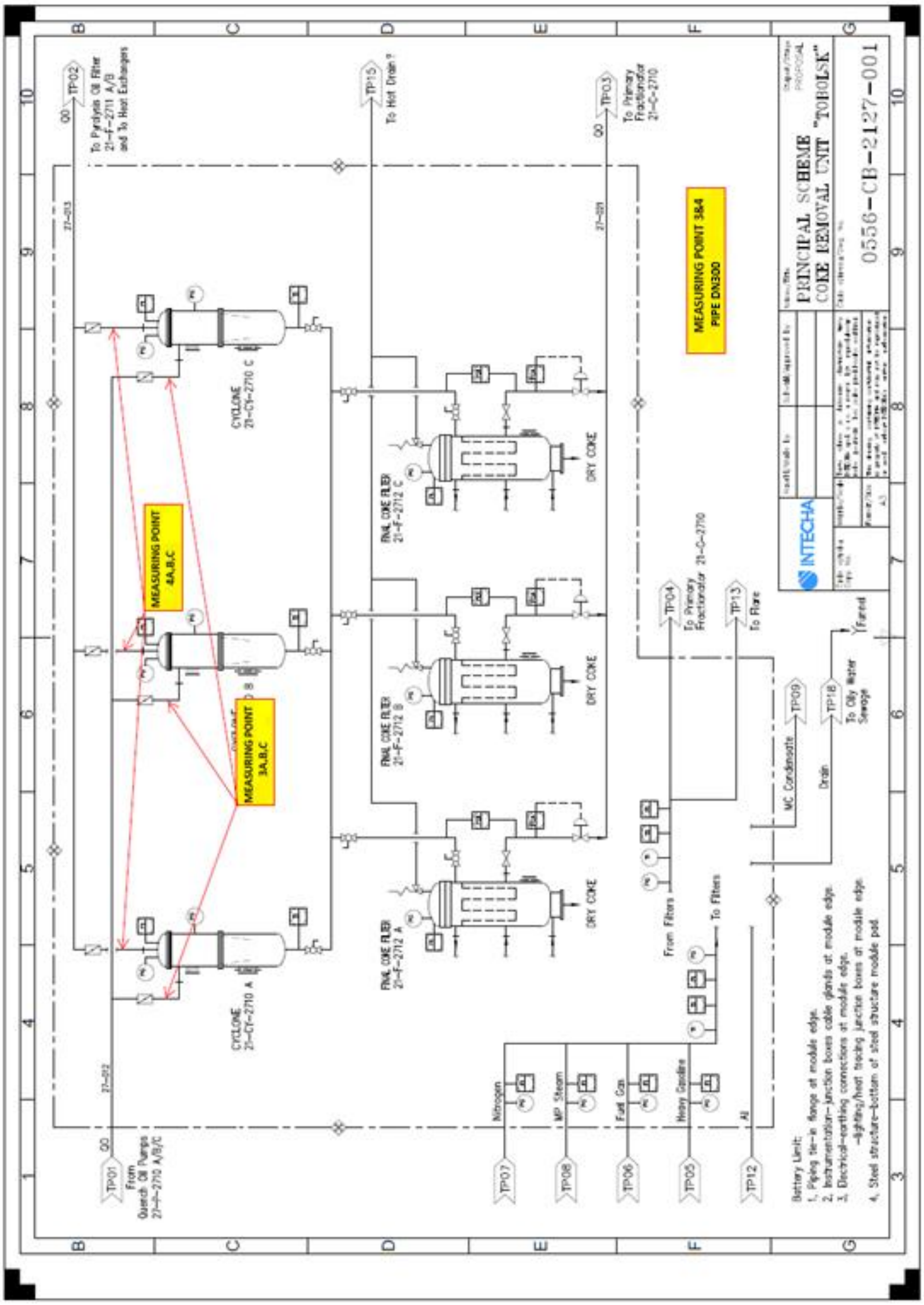


Figure 13: Process setup

5. LABORATORY EXPERIMENT

For laboratory experiment is the same device (Oil in water, water in oil, multiphase oil, produced water, tar sands measurement systems, attachment [2]) as for process measuring only with different process connection size to fit on the pipe DN25 PN6 (model VM6C111BF3V).

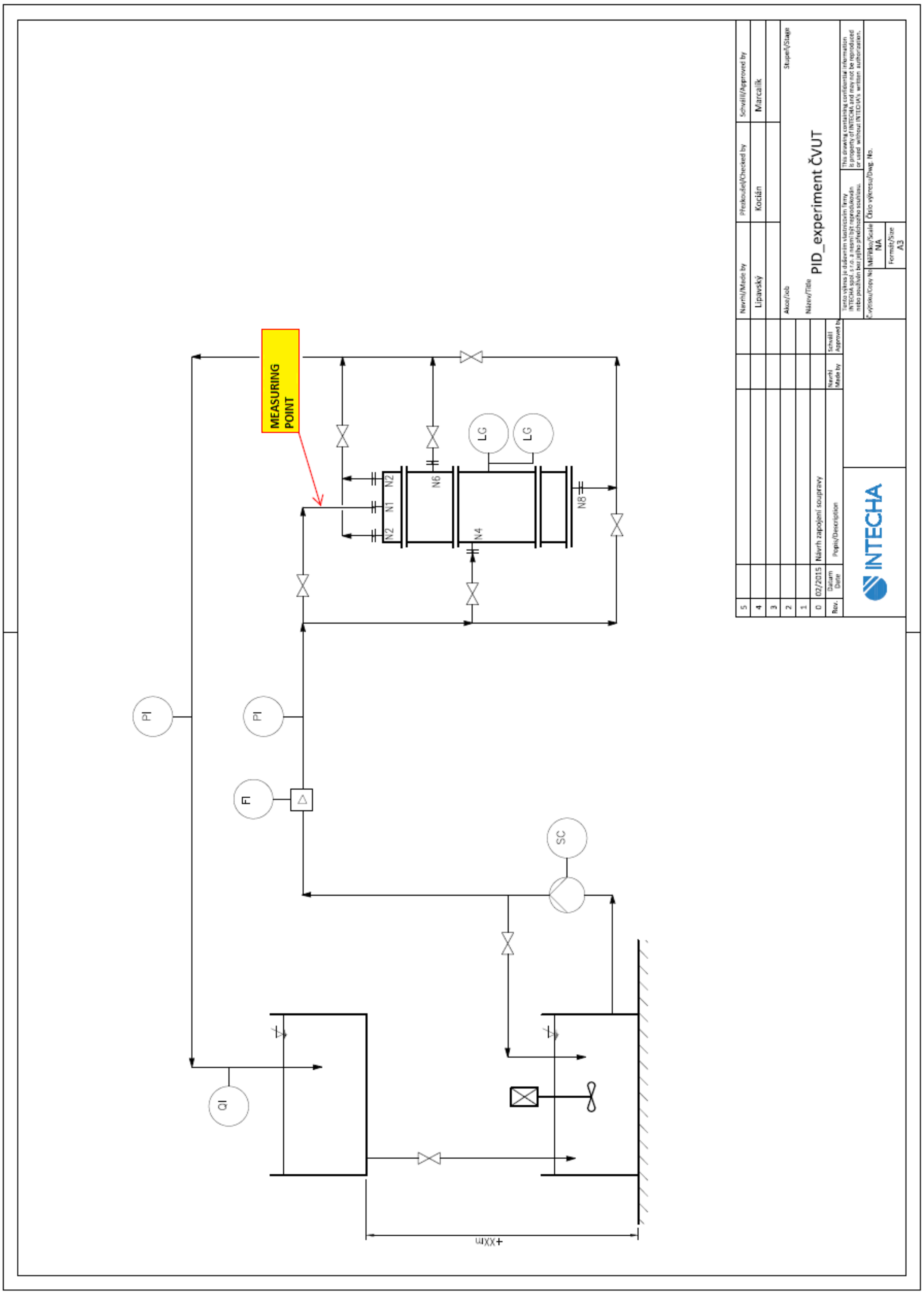


Figure 14: Experimental setup

6. CONCLUSION

On-line analysis allows mal-operation of many different unit operations to be more rapidly identified thereby reducing plant downtime and increasing unit productivity. Successful particle size analysis relies on each element of the analytical package being appropriate for the application.

While searching for possible device for measuring solid particles in liquid medium, the only possible on – line technologies for use in industrial field were based on optical methods. Each company has different scanning system and every system has its own limitations and preferable use. When purchasing a device there are many parameters to be considered in order to have trustworthy output. The main criteria is particle concentration and size, medium transparency and medium characteristic such as temperature, flow rate or pressure. Another important criteria is if medium is well mixed and homogenous, so scanning could be done by probe or bypass, or if there is a need to scan whole volume to achieve reliable particle analysis.

In this case I chose device “Oil in water, water in oil, multiphase oil, produced water, tar sands measurement systems” from Canty company with high output light and CCD camera. Light allows to measure dark oils and camera system together with software is able to recognise and evaluate oil drops and solid particles in water and in oil, even with small particle concentration. It is flanged system with connection size that fits to proposed measuring point and it scans whole volume of the pipe. Design also reflects other parameters like high pressure and temperature or requirement for measuring in hazardous environments.

Despite higher purchase price of on-line analysis, this method with correct configuration might be more reliable and more profitable in the long term use than laboratory sampling.

7. LIST OF FIGURES

8. LIST OF ATTACHMENTS

[1] Table of parameters

[2] „Oil in water, water in oil, multiphase oil, produced water, tar sands measurement systems” datasheet

9. REFERENCES

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