CZECH TECHNICAL UNIVERSITY IN PRAGUE

FACULTY OF MECHANICAL ENGINEERING
DEPARTMENT OF PROCESS ENGINEERING

BACHELOR THESIS

AUTOMATIC CHARACTERIZATION OF PARTICULATE MATTER BY SEDIMENTATION

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BACHELOR‘S THESIS ASSIGNMENT

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Study program: **Bachelor of Mechanical Engineering**
Branch of study: **Power and Process Technology**

II. Bachelor’s thesis details

Bachelor’s thesis title in English:

Automatic characterization of particulate matter by sedimentation

Bachelor’s thesis title in Czech:

Automatická charakterizace partikulárních látek pomocí sedimentace

Guidelines:

The theoretical part of the work will focus on the sedimentation of concentrated suspensions, including the basics of image analysis. In the practical part, recordings of the sedimentation tests of calibration and real samples of particulate matter will be made. The recordings will then be divided into frames. The frames will be subjected to image analysis in order to find the connection of the frames difference with the sedimentation curve. The image analysis procedure will be automated in Matlab. The work will include a standard for recording of a sedimentation test and an algorithm for automatic processing and evaluation of the recording. The aim is the automatic characterization of particulate matter by sedimentation.

Bibliography / sources:

1) ISO 12103:2016 Road vehicles — Test contaminants for filter evaluation; 2) ALLEN, Terence. Powder Sampling and Particle Size Determination; 3) Matlab support documentation - Image Processing Toolbox, ROI, Pixel brightness histograms

Name and workplace of bachelor’s thesis supervisor:

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III. Assignment receipt

The student acknowledges that the bachelor’s thesis is an individual work. The student must produce his thesis without the assistance of others, with the exception of provided consultations. Within the bachelor’s thesis, the author must state the names of consultants and include a list of references.

.	
Date of assignment receipt	Student’s signature

Declaration:

I confirm that the bachelor’s work was disposed by myself and independently, under leading of my thesis supervisor. I stated all sources of the documents and literature.

In Prague …05.08.2022…………… …………………

. Name and Surname

Acknowledgement:

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Annotation sheet

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Abstract

This thesis deals with the automatic characterization of particulate matter by sedimentation. The thesis covers the Description of particulate matter, particle size distribution, sedimentation, sedimentation of suspended particles, sedimentation curve including the basis of optical analysis. This knowledge will be used to design and measure road dust samples for classification. The output of this work is a standardized procedure suitable for field conditions for the characterization of road dust according to the standard ISO 12103-1

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List of abbreviations:

Ar	Archimedes number
u	Sedimentation rate of suspension
T	Temperature
t	Time
n	Particle dispersion parameter
g	Gravitational acceleration
µ	Dynamic viscosity
C_D	Drag coefficient
D	Stokes diameter
ρ_s	Density of solid
ρ_l	Density of liquid
Re	Reynold’s number
U_0	Fall rate of sedimentation
T_{ch}	Characteristic time
μ_m	Viscosity of mixture
Ψ_p	Empirical correction factor
ρ_m	Bulk density
c_{max}	Maximum concentration
$\mathcal{E}$	Porosity
C	Volume concentration of particles
d	Particle diameter
D	Container diameter
ISO	International standard organization
RGB	Red, Green, Blue
PM	Particulate matter

1. Theoretical Part

1.1 Introduction

The industrialization of society requires the expansion and urbanization of road systems. The formation of street dust in urban areas is the result of industrialization. Road dust is composed of solid particles produced by the mechanical processing of materials, including the crushing, explosion, and decomposition of organic and inorganic materials such as rocks, ores, and metals.[1] if this dust floats primarily due to friction of tires travelling on unpaved or dust-covered paved roads. It is known as road dust.

Risk conditions when handling samples dust occurs when the respiratory system is exposed to particulate dust by air.it is recommended to use a respirator or dust mask approved in accordance with national regulations.

Road dust can also be found in the mining industry. Road dust found in mining areas consists of dust from multiple sources, including wind-blown mineral dust and topsoil from mines, and uncovered truck leaks. They are then distributed via wind and traffic activities and become an important source of particulate matter (PM).

Dust control is a serious problem in an industrial work environment for to following reasons:

- Dust can damage expensive manufacturing equipment’s and slow down overall operation.
- Dust can obstruct the view of workers and cause occupational accidents.
- Dust leads to high cleaning costs, both inside and outside of the facility.

1.2 Objective

The main objective of this work is to automatic characterization of the road dust according to ISO 12103-1. standards and develop a standardized procedure for the road dust samples and perform image processing on sedimentation test video in MATLAB.

1.3 Description of particulate system

Process engineering is primarily concerned with the description of particulate system, i.e., systems containing solid substances in the form of particles. At first the idea can be that such systems can be considered as a continuum in a sufficiently macroscopic view, and that they could therefore be described by the same laws. However, practice shows that it can be otherwise. An example is an hourglass. From any device for measuring time, a constancy of reading is expected for successful use. If we were to take the sand in hours as a continuum, then the flow through the slot would depend on the height of the column of liquid above the slot. In fact, with an hourglass, the mass flow of sand is almost constant, which eliminates the connection with the idea of continuum. The pressure for bulk materials does not increase linearly with the height of the column as for liquids, but after a certain rise time it almost stabilize, allowing the use and existence of hourglass. This happens because of the friction between the individual particles and because of the friction between the particles and the wall of the container. moreover, this interparticle force is inversely proportional to the size of the particles.[2] thus, the process taking place in particulate systems cannot be analysed only based on the external forces, but also necessarily to consider the mutual interactions of the particles. In general, particulate systems can be characterised using the following:

- Chemical composition
- Description of the dispersion component, i.e., particles
- Mutual force action between particles and force action at the phase interface
- Other state variables

For the context of this work, the description of the dispersion component of the particulate system, partly also the mutual force interaction between the particles is a crucial point. Further particle systems are divided according to the size of the particles:

- Molecular dispersion systems – particle size smaller than 10^{-9} m.
- Colloidal dispersion systems – particle size in between 10^{-9} – 10^{-6} m.
- Coarse dispersion system – particle size greater than 10^{-6} m.

The last mention category is relevant for the content of this work, that is particles larger than 1 micron. The very behaviour of particulate systems is mostly dependent on the morphology and size of the individual particles as well as their distribution. there is an abundance of methods for determining these characteristics. However, it must be kept in mind that a relevant comparison of different results is usually only possible with data that was obtained using the same measurement method. For maximum comparability ideally on the same device with the same operator. Particle description can be relatively simple discipline in the case of spherical particles. In the case of non – spherical particles, the situation is considerably more complicated. Over time tools were invented to describe non regular particles. These are often methods where an irregular particle is transformed into an equivalent sphere according to selected parameters, for example the same volume. These equivalent averages can already be considered for subsequent calculations.

1.4 Particle Size Distribution

Assuming that all particle systems are monodisperse and the particles in them are the same, a single value such as diameter is sufficient to represent the size of the particles. Such systems are minimal, so you need to choose a description that properly characterizes the particle system. You can use various statistical indicators, such as the arithmetic mean. This is perfectly legitimate, provided that the particles are at least partially monodisperse and the mean is not misleading. In order to convince the properties of the particle system, it is necessary to draw a so-called distribution curve. In general, the distribution curve can be characterized as the distribution of the abundance of particle numbers on the z-axis for a particular statistical weight plotted on the y-axis. Depending on the statistical weighting selected, the distribution curves fall into five categories and are described in Table 1. It is noteworthy that the method of measuring the distribution curve at the same time determines the type of distribution obtained. The index r is used to accurately determine the statistical weights of the distribution curve.

Statistical Weight	Dimension	Label with Index
Number	L^0	r = 0
Length	L^1	r = 1
Area	L^2	r = 2
Volume	L^3	r = 3
Mass	L^{3*}	r = 3*

Table 1: Statistical Weights

Index 0, number weighted distribution particles are counted in individual size groups with a delta x size step defined before the data analysis. All count measurement methods generate this type of distribution. in general statistics the number is an almost exclusive weight, the numbers in individual groups are then called relative frequency.

Index 1, length weighted distribution, this rather rare distribution is typically used for particles where one dimension significantly exceeds the other dimensions. These are usually narrow, elongated needle-shaped particles.

Index 2, area weighted distribution this is either the actual surface of the particles or the easier to obtain projection surface of the particles again arranged into size groups. Most often, this distribution is obtained using optical measurement methods.

Index 3, volume, or weight-weighted distribution values reported in individual size groups are obtained by weighting for the weight-weighted interface. For the case where the particle density is independent of their size, the mass-weighted distribution corresponds to the volume-weighted distribution.

The distribution curve of the particles is the only display that can clearly determine whether the investigated particulate system is monodisperse, bidisperse or polydisperse. The course of the distribution curves for different statistical weights is different and the choice of the right curve depends on the purpose of use. If the essential factor for the course of the technology is the volume or weight of the particles, it is necessary to use the corresponding curve. If necessary, a recalculation between individual statistical weights can also be performed using so called moments.

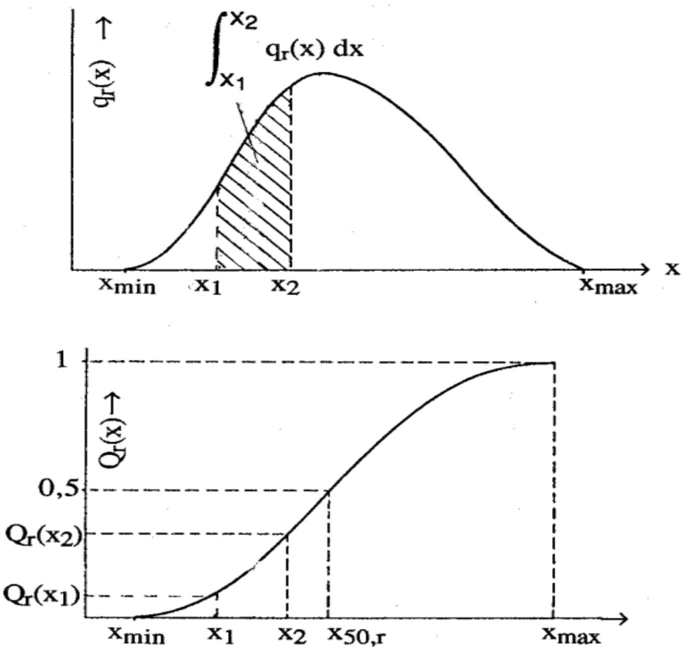

Figure 1: The relationship between integral and differential distribution curves

Particle size distribution curve with the chosen statistical weight can have several forms. They can be represented discretely by means of histograms, alternatively continuously in the form of probability distribution q(x), alternatively by the sum function Q(x). let a collection of particles of different sizes be given, for simplification spherical aligned according to the diameter, which is to be sorted into individual size groups. A graph is drawn where the size of the particles x (equivalent diameter for non-spherical particles) with the width of the interval Δx is plotted on the x-axis. The length of the step can be chosen as a proportion of the total distribution width (maximum number of occurring particles) divided by the square root of the number of measured particles.[5]

$$\Delta x_i = x_i - x_{i-1} \tag{1}$$

Individual intervals i bear the designation according to the upper limit of the interval, that is the interval with the boundaries i and i+1 will be designated as interval i+1 according to the generally accepted convention. The second option is to mark individual size classes with mean value. For distributions with narrow intervals, the pro their identification uses the arithmetic average of the particle sizes at the boundaries of the intervals (see equation 2), in the case of distributions with wider intervals, in exceptional cases, the geometric mean can also be found calculated from the particle size at the boundaries of individual intervals (see equation 3). [3]

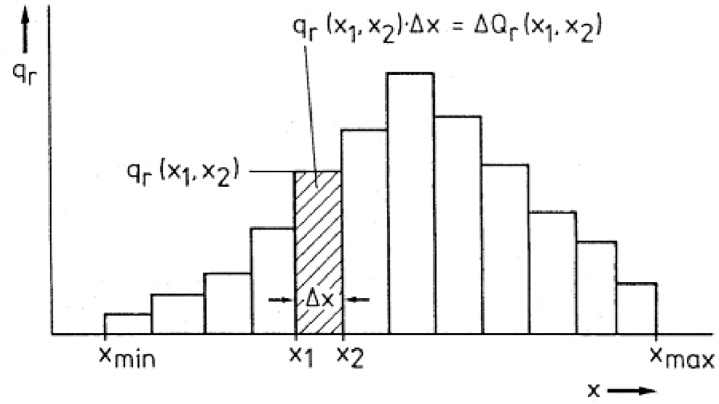

Figure 2: Differential distribution curve in the form of histogram [3]

$$\overline{x_i} = \frac{x_1 + x_{i+1}}{2} \tag{2}$$

$$\bar{\bar{x}}_i = \sqrt{x_1 * x_{i+1}} \tag{3}$$

The proportion of the total amount of the given statistical weight that are smaller than the particle size of the given interval is then plotted on the y-axis. For a better understanding, the following example with a number weighted distribution is used. Let the particle interval described by the mean value (10µm) be described. The number of particles smaller than the characteristics size of the interval (10µm) divided by the total number of particles must then be plotted on the y-axis. Similarly, the y- values for the remaining intervals are plotted. This procedure yields the so-called sum fraction Q(x) (see figure2). Already from the definition of the sum function, it is obvious that the course of the function will increase with the increasing size of the particles on the x-axis, or it will be constant, but it can never decrease. If the sum function is divided by the width of the interval the probability distribution curve q(x) is obtained. [3]

$$q_{r,i}(x) = \frac{Part\ of\ the\ quantity\ in\ the\ interval\ i}{Total\ amount * The\ width\ of\ the\ interval} \tag{4}$$

1.5 Sedimentation

Sedimentation is one of the basic ways of separating solids and liquids. It has won its place not only in history but mainly thanks to its unity. In general, sedimentation could be described as the movement of solid particles in a liquid from the higher energy layer to the lower one. This movement can be caused by gravitational or possibly centrifugal forces. This is of course, under the assumption that the particles will have a larger skeleton density than the density of liquid.[6]

However, sedimentation does not have to serve only as a separation process, but also as an analytical method. It thus dated back to the middle of 19th century, when George Gabriel Stokes published the solution of the Navier-Stokes equation for small Reynolds numbers for the movement of a spherical particle, which is followed by the stokes equation, which already indicated the relationship between the sedimentation velocity and particle size. For non – spherical particles, the output is the Stokes equivalent diameter.[6]

Force Balance in Sedimentation

The behaviour of a particle falling through a liquid in the Earth's gravitational field is evaluated based on the balance of forces. In order to consider the balance of forces, it is necessary to consider that the movement of particles is uniform, that is at a constant velocity. For solubility, incompressible spheres are considered as particle.

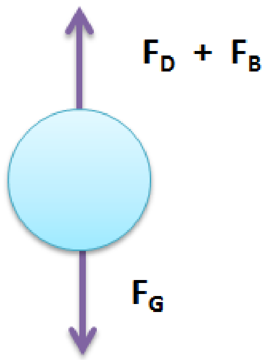

Figure 3: Force balance on a spherical particle

The following forces acts on the particle:

Drag Force: $F_D = C_D * \frac{\pi D^3}{6} * \rho_s * \frac{u^2}{2}$

Gravitational Force: $F_g = \frac{\pi D^3}{6} * \rho_s * g$

Buoyant Force: $F_b = \frac{\pi D^3}{6} * \rho_L * g$

To fulfil the condition of the constant velocity of the particle, the following equation must apply:

$$F_D + F_b - F_g = 0 \tag{5}$$

The only obstacle in specifying the settling velocity or particle shape is the determination of the drag coefficient C_D included in the drag statement. This is a function of the Reynolds number Re of the particle, and therefore the velocity of particle itself.

$$Re = \frac{\rho_{L*} u_0 * D}{\mu} \tag{6}$$

The drag force can be divided into two components. When a viscous liquid flows slowly around the bod, in the case of liquid settling, it is assumed that the liquid attaches to the walls of the body in the flow. Therefore, the shear stress on the wall is not zero, and the force acting on the body is not zero. The second element of drag is the pressure that is unevenly distributed around the body which also causes non-zero forces.

The C_D course Re dependence (see Fig.4) can be divided into three parts. After the drag coefficient first linearly decreases in the range of approximately $R_e < 1$ this range corresponds to the validity of Stoke's law, the so-called Stokes range. In this area the main element of drag is the action of viscous shear forces. This range is essential for sedimentation and Stokes adds the following relationship. It can already be used to calculate the velocity of the particle or its size. (See equation 6)

$$C_d = \frac{24}{Re} \tag{7}$$

This is followed by so called transition region, where the curve declines more slowly up to a value of approximately Reynolds number Re = 500. This is followed by the Newtonian region, where the drag coefficient can be considered as a constant. In the Newton region, in contrast to Stokes region the main component of the force is no longer the viscous. As the Reynold number increase further, a sharp decrease occurs in a narrow region around $Re = 4 * 10^5$ to step reduction values of the resistance coefficient.

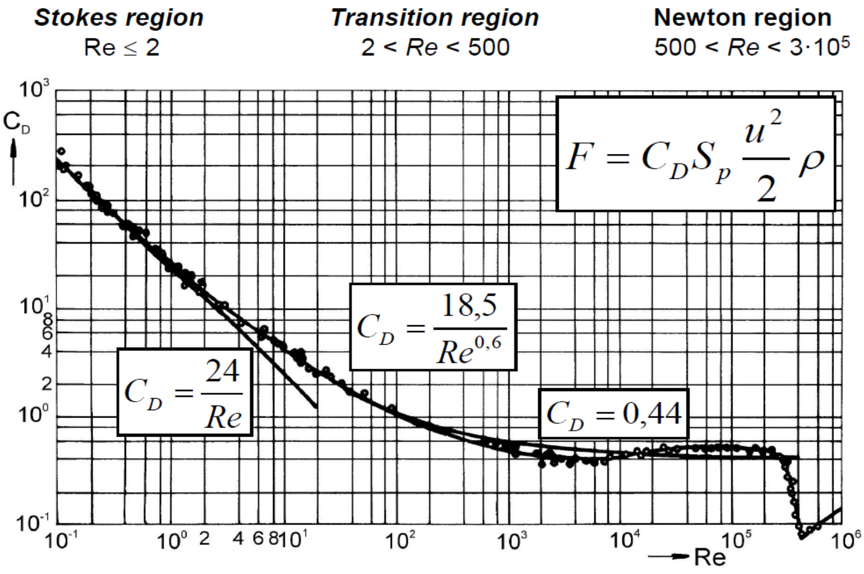

Figure 4: Dependence of C_D and R_e for spherical particle [4]

This decrease is known as a pore crisis and is associated with change in the body's coverings. As already mentioned, being in the Stokes area is advantageous for analytical sedimentation. Therefore, if the particles slowly surrounded or are small, they will probably occur at a lower particle density. Another option is a high viscosity value of the selected fluid. The output in this range to the Stokes sedimentation velocity which is called Stokes equation.

$$u = \frac{\Delta\rho g}{18\mu} D^2 \tag{8}$$

where D is the Stokes diameter, Δρ is the difference between the densities of the particle skeleton and the liquid, μ is the dynamic viscosity of the liquid, g is the gravitational acceleration and u is the stokes sedimentation rate. In his derivation Stokes considered three forces acting on a particle: gravitational, buoyancy and drag force. So, he neglected the influence of the inertia of other particles, the effect of the wall or the free surface as well as the inertial force. Despite the neglect of some of the factors mentioned above the error for calculating the stokes velocity should not exceed 5%. For even smaller Reynolds numbers less than 0.2 the error should be kept within 0.5%.[12]

In the case of analytical sedimentation to determine the Stokes diameter of the particle, it is not impossible to verify whether the experiment is being conducted in Stokes region (see fig. 4). To see the range, you need to know exactly what the unknown diameter is being sought. One of the possible solutions is to use the Stokes relation in the calculation, and use the average obtained to check the Reynolds number to justify the previous method. If the resulting diameter is in a different range, you can adjust the working conditions (the viscosity of the liquid) or consider another method pf particle analysis. The uniform movement of the particle was described in the previous lines. However, when explaining the actual experiment, it is assumed that the particle has zero velocity at time t = 0. Therefore, the question arises whether the acceleration of the particle from zero to the Stokes velocity can basically influence the process of measurement. The effect can be determined retrospectively after determining the size of the particle. The particle approaches the constant velocity exponentially and after substituting into equation 9, we obtain the relaxation time, which is the characteristics time when the exponential approaches the stationary state.

$$t_{ch} = \frac{\rho_s * D^2}{18\,\eta_l} \tag{9}$$

For a time value five times greater than the characteristics time, the deviation from the constant speed is less than 1%, which can be reasonably accurate.[13] During the time, the distance covered will then be:

$$y_{(5tch)} = \frac{Ar * \rho_S}{81\rho L} D \tag{10}$$

$$Ar = C_D * R_e = \frac{g * d^3 * \rho_L * (\rho_S - \rho_L)}{\mu^2} \tag{11}$$

To estimate the effect of the acceleration, a typical spherical particle with a size of 10µm and density of 2650 $\frac{Kg}{m^3}$ and sedimenting in ethanol at 20°C. for a particle of this size, the characteristics time is of the order of t_{ch} = 10^{-5} and subsequently the distance travelled for the value of five times the characteristic time y ($5t_{ch}$) = $6.\ 10^{-12}\ m.$ After calculating these values, it can be said that the effect of particle acceleration is negligible under certain conditions described in the experimental part.

1.5.1 Sedimentation of concentrated suspension particles

In the previous part of the thesis, the assumptions were used to determine the sedimentation behaviour of particles (determination of sedimentation speed). The liquid was taken as a continuum with Newtonian behaviour the influence of the walls and other particles was not considered. Due to the focus of this work, in the following paragraphs the author focused on the effect of higher concentration on the course of sedimentation.

For suspensions with a volume concentration greater than 0.2% [3] the influence of other particles is already negligible (the Stokes shape of the sedimentation velocity does not apply) and it is reasonable to talk about sedimentation in the hindered settling. The change in particle behaviour at higher concentrations occurs due to mainly three reasons:

1. When a particle falls through a liquid, it may encounter another particle resulting in a change in trajectory. The probability of collision increases with increasing particle concentration
2. Another reason is liquid backflow. During sedimentation, not only the particle sinks but also part of the liquid in its immediate vicinity. As part of the validity of the continuity equation, a flow of liquid must also occur upwards, the so-called backflow. As a particle concentration increases, this reverse flow increases proportionally, acts against the sedimentation particles, and thus slow down the sedimentation process.
3. Mutual electrostatic action in case of deposition of charged particles.

Given that the volume occupied by the particles, and not their weight, is essential for the above-mentioned point 1 and 2. The volume concentration of the particle is suitable parameter describing disturbed settling. The investigated samples from the experimental part of the work are mostly composed of silicon dioxide and other elements (see table 4). Due to the nature of the investigated dust and the way it is handled, the deposition of charged particles is not expected, and therefore this issue will not be further elaborated in the theoretical part of the work.[13]

1.5.2 Hindered Settling

For many cases of settling, a large number of particles are present, and the surrounding particles interfere with the motion of individual particles. The velocity gradients surroundings each particle is affected by the close presence of other particles. The particles settling in the liquid displace the liquid and an appreciable upward velocity of the liquid is generated. Hence the velocity of the liquid is appreciable greater with respect to the particle than with respect to the apparatus itself.

For such hindered, flow the settling velocity is less than the calculated from equation 4 for Stoke's law. The true drag force if greater in the suspension because of the interference of the other particles. This higher effective viscosity of the mixture μ_m is equal to the actual viscosity of the liquid itself μ, divided by an empirical correction factor Ψ_p, which depends upon $\mathcal{E}$, the volume fraction of the slurry mixture occupied by the liquid [12]

$$\mu_m = \frac{\mu}{\Psi_p} \tag{12}$$

Where Ψ_p is dimensionless and is as follows:

$$\Psi_p = \frac{1}{10^{1.82(1-\mathcal{E})}} \tag{13}$$

The density of the fluid phase effectively becomes the bulk density of the slurry ρ_m, which is:

$$\rho_m = \mathcal{E}\rho + (1 - \mathcal{E}) * \rho_p \tag{14}$$

Settling velocity v_t with respect to the apparatus is $\mathcal{E}$ times the velocity calculated by equation 8.

When the Reynolds number is less than 1, the settling is in the Stoke's range. For Reynolds number above 1 the effect of concentration is greater for non-spherical particles and angular particles.

1.5.3 Sedimentation curve

As the concentration increases, a sharp interface is gradually formed between the sedimenting particle and the clear liquid. A sedimentation curve is created by plotting the decrease in the height of this stage as a function of time.

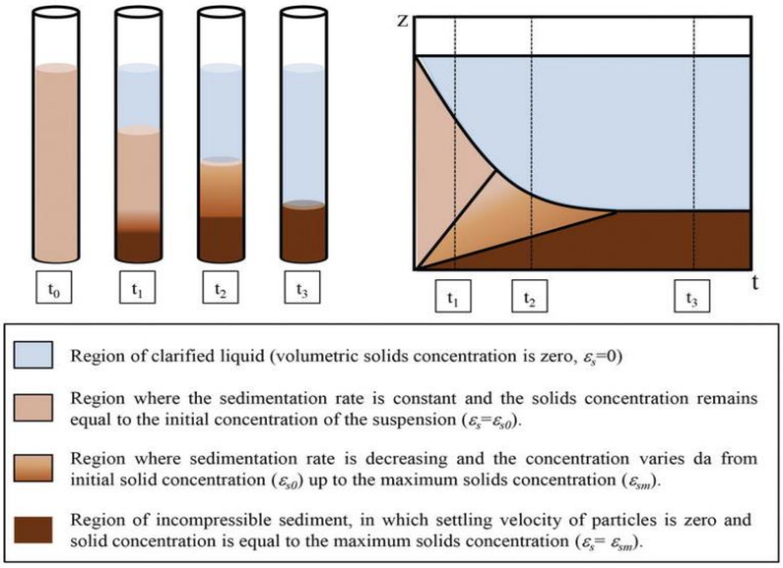

Figure 5: Individual phase of sedimentation according to Kynch's theory [5]

Well watchable the interface facilitates the reading of its height during manual measurement during the experiment and thus contribute to more accurate results. A well observable interface was reached in the case if Arizona dusts at concentration above approximately 6% by volume. This value depends on the dust concentration, as the coarseness of the dust increases, so does the optimal concentration. The course of the sedimentation curve is plotted in Fig 5. It assumes that the suspension was properly homogenized before the start of the experiment, so that the starting conditions are not conditioned by local differences in the concentration of solid particles. It goes without saying that handling the suspension should not lead to the formation of bubbles in the suspension, which would be assessed by sound measurement. The course of the sedimentation curve can be divided into three parts. [5] The 1st area (marked as I in Fig 5) is an area of constant sedimentation rate, so the course of the sedimentation curve is linear. This region is essential for determining the sedimentation rate of the particle cloud by manual optical reading of the interface height drop. In certain cases, a start-up phase can be observed, when the approach to the linear part occurs. The measured points of this run up phase could bias the particle size evaluation and should not be used for evaluation. As the concentration increases, the curve flattens, and the sedimentation process takes place more slowly. During the transition to region II, the suspension begins to thicken from the original concentration C_0 and the velocity of the particle decreases. The gradual increase in the concentration of the suspension is graded by the transition to region III, where the sediment is finally compresses by its own weight until maximum possible concentration C_{max} is reached, while the particle velocity is also zero. When comparing the maximum volume concentration and porosity, the following relationship applies: [7]

$$C_{v,max} = 1 - \mathcal{E} \qquad (15)$$

In some cases of particles with a broad distribution curve, multiple interfaces may occur.[19] because of the backflow, the smallest particles are washed above the main part of the smallest particles are washed above the main part of the suspension. This creates a secondary suspension of very fine particles, which create one or more additional interfaces above the main suspension.

1.6 Optical Analysis

For this work, the basics of optical analysis of particle systems are briefly explained in the next line. Optical measurement (image processing) has undeniable advantages over other analytical methods. An unknown sample of the particle system can be used to estimate the size of the particles and explain the tendency of the system to form clusters, the shape of the particles, and the properties of the surface. In general, a form in which the next intervention can be decided immediately on the screen. Therefore, this should be the first step in analysing an unknown particle sample. As far as the numbers are concerned, most optical techniques are important. That is a numerically weighted distribution curve is created. If you need to change the statistical weights, you need to recalculate the distribution curve using statistical techniques.

For the optical analysis in the context of this work was done by Image processing toolbox in MATLAB. The name MATLAB consists of two words matrix and laboratory. According to MathWorks (the manufacture of MATLAB), MATLAB is a technical computing language that is primarily used for powerful numerical calculations and visualization. Integrate data processing, programming, signal processing and graphics into a user-friendly environment to express problems and solutions using mathematical notation. The basic data element is an array that allows the calculations of the difficult formula which is mainly found in linear algebra. But MATLAB isn't just about math problems. It can be widely used for data analysis, modelling, simulation, and statics. MATLAB's advanced programming languages have applications in other scientific disciplines. [14]

Image processing is am method of performing some operations on an image to get an expanded image or extract some useful information from the image. This is a type of signal processing where the input may be image and the output may be an image or a property/function associated with that image. The image processing toolbox is a collection of functions that extend the capability of the MATLAB numeric computing environment. The toolbox supports a wide range of image processing operations, including

- Spatial image transformations
- Morphological operations
- Neighbourhood and block operations
- Transforms
- Image analysis and enhancement
- Image registration
- Deblurring
- Region of Interest operations

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From the above-mentioned image processing operations, the Transforms and the Region of interest operations explained below.

Transforms is the usual mathematical representation of an image is a function of two spatial variables f(x,y). the value of the function at a given position (x,y) represents the intensity of the image at that point. This is called the spatial domain. The term transformation refers to another mathematical representation of an image. For example, the Fourier transform is a representation of an image as a sum of complex exponentials of various sizes, frequencies, and phases. This is called the frequency range. [15]

The Fourier transform is a representation of an image as the sum of complex exponentials of various sizes, frequencies, and phases. The Fourier transform plays an important role in a wide range of image processing applications such as enhancement, analysis, restoration, and compression. If the f (m, n) is a function of two discreate spatial variables m and n then the two-dimensional Fourier transform of f (m, n) is defined by the equation 16.

$$\mathrm{F}\ (\omega_1\omega_2) = \sum_{m=-\infty}^{\infty}\sum_{n=-\infty}^{\infty} f\ (m,n)e^{-j\omega 1m}e^{-j\omega 2n}d\omega 1 d\omega 2 \qquad (16)$$

The variable ω1 and ω2 are frequency variables. The unit is radians per sample. F (ω1, ω2) is often referred to as the frequency domain representation of f (m,n). F (ω1, ω2) is a periodic complex valued function with ω1 and ω2 with a period of 2π. Due to its periodicity, only range $-\pi \leq \omega 1, \omega 2 \leq \pi$ is displayed.

Region of Interest (ROI) is a portion of an image that you filter or edit in some way. You can display the ROI as a binary mask image. Pixels belongings to the ROI are set to 1 and pixels outside the ROI are set to 0. ROI can consist of groups of contiguous and discontiguous pixels. A contiguous area is one group of connected pixels. Consecutive ROI can represent a single object in an image. Cars in street scene image or body tissue in medical images. For example, an individual ROI can represent every pixel that corresponds to water in an aerial image. Image processing toolbox provides many options to create a binary mask. You can create mask using single threshold. The ROI consists of grayscale pixels whose intensity is above (or below) a specified threshold. One can create a binary mask representing the ROI using the function called imbinarize.[15]

Figure 6: Binary mast of the flower [15]

You can also create mask based on the position. The ROI consists of all the pixels whose position is within a geometrical or a hand drawn shape. First, you can create ROI object then you create a binary mask using the function createMask. You can also create ROIs of various shapes, including circles, ellipses, polygons, lines, polylines, rectangle, and hand drawn shapes.

For this work the ROI of the rectangle shape was created.

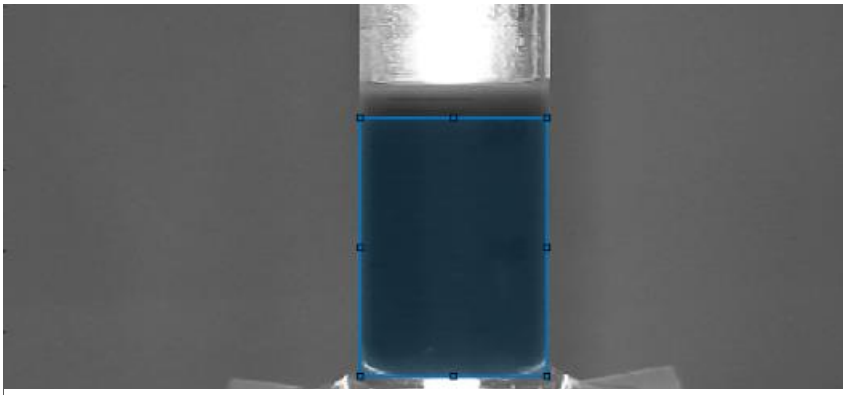


Figure 7: Rectangle ROI

As you can see in the figure 7 for region of interest, we only need that part of image which is important for sedimentation due to that we only consider the rectangular part where sedimentation happens.

2 Experiment Part

Four samples of dust were supplied for the identification of the particulate dust. In this work characteristic diameter of the set of particles were determined based on the sedimentation test.

The aim of the experimental part is to measure the characteristics of Arizona dust of the possibilities of classification of collected dust samples into individual categories based on their particle size distribution. And do an automatic characterization of sedimentation using the image processing toolbox in MATLAB.

2.1 Characteristics of Arizona dust according to ISO 12 103

- The standard gives the dust density of 2650 kg.m^{-3}.
- Chemical composition listed in Table

Chemical	Mass fraction [%]
SiO_2	68 to 76
Al_2O_3	10 to 15
Fe_2O_3	2 to 5
Na_2O	2 to 4
CaO	2 to 5
MgO	1 to 2
TiO_2	0.5 to 1
K_2O	2 to 5

Table 2: Chemical Composition of Arizona dust

2.2 Sedimentation Test

Sedimentation test for dust analysis uses a linear decrease of the suspension interface – clarified liquid in the first phase of sedimentation (see the Fig. 8). By determining the direction of the straight part, the sedimentation rate of the suspension is determined in the height – time diagram. For graphical comparability of the two sedimentation tests of the same concentration the graphs were plotted with no dimensional height H/H_0 on the y-axis where H is the initial height and H_0 is the interface height. It should be kept in mind that only sedimentation tests with the same volume concentrations of the particles can be compared graphically.

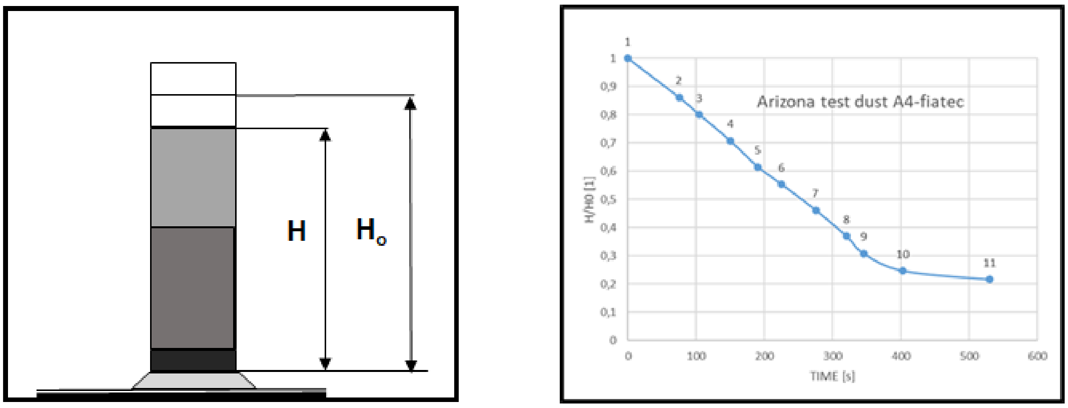


Figure 8: The course of sedimentation and shape of sedimentation curve

2.3 Sedimentation Test Procedure

For sedimentation test the 50 ml cylinders were used. The standard liquid used was 96% of the available ethanol.

- Only a dry cylinder can be used
- The Apparatus is an empty cylinder
- Measure 30 ml of ethanol in an auxiliary graduated cylinder (or any other amount to maintain optimal concentration) which is poured through a funnel into the graduated cylinder.

- The required amount of dust is poured into the cylinder placed on the balance using a spoon.
- Homogenize the suspension with a homogenizer as gently as possible and the particle will begin to rise.
- When homogenization is complete the camera will start.
- Interface height readings decrease over time as the level stabilizes in the test.

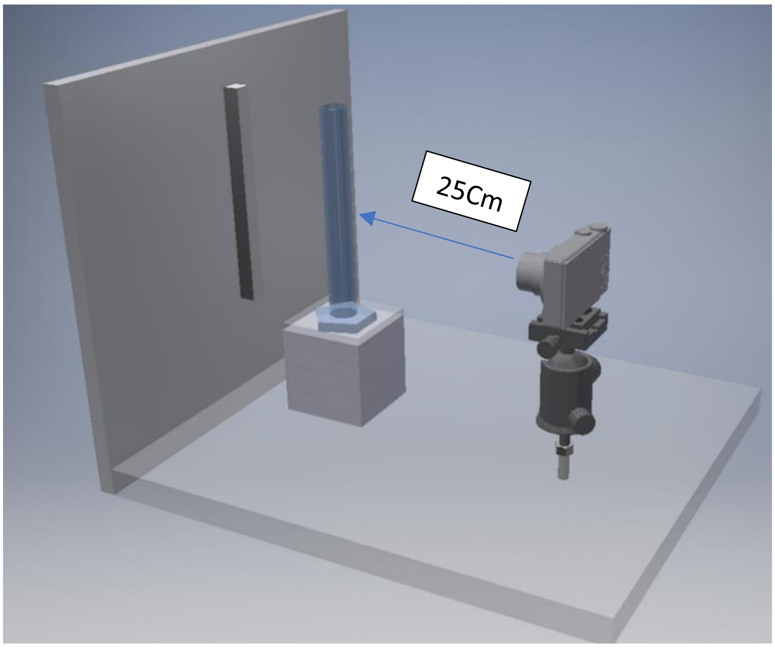


Figure 9: Set up for sedimentation test

- When the level stabilizes, the test ends.

Preparation

The preparation for the sedimentation tests (see Figure.9). The supporting jig of the product is made of plastic - ABS. The measuring cylinder is illuminated by an LED lightning system so that it does not dazzle the sensor (camera / mobile phone) located on the tripod head. Surrounding around the set up was standardly lit room and the temperature of the room was 25°C.

2.4 Camera setup

- Movie size: 1280 x 720 (Fine) pixel
- Zoom – manual use mode
- Exposure compensation = 0
- Standard Image brightness = 0
- White balance - Incandescent
- Movie steady shot – Standard (under moderate movement)

2.5 Sedimentation Test Protocol

Input Data:

- Temperature
- Ethanol volume (standard 30ml) with 96% concentration
- Calibration cylinder gauge
- Dust skeletal Density
- Dust sample weight
- Decrease in the height of the interface over time.

The outputs of the protocol after completing the sedimentation curve estimate are:

- Sedimentation rate of the suspension
- Sedimentation rate of representative particle
- Used dust concentration
- Recommended dust class category
- Graphical representation of the course of sedimentation recalculated using the relation (equation number) to a volume concentration of 9%, to enable rapid optical classification of dust into one of four categories. the conversion is only valid for Zaki's relation validity area. That is the range of 5 to 10.5%.

In the background of the protocol, calculations are carried out to determine the appropriate coefficient for the Richardson and Zaki equation, the calculation is iterative, the first value is the standard n = 4.65. further the ethanol viscosity is corrected for the specified measurement temperature. Another calculation is recalculation of the measured the sedimentation curve. when the curve is recalculated to 9%, which subsequently enables classification based on comparison with the course of the sedimentation curve of standard dusts.

$$\frac{4.8-n}{n-2.4} = 0.0365Ar^{0.57}\left[1 - 1.24\left(\frac{d}{D}\right)^{0.27}\right] \tag{17}$$

Where Ar is Archimedes number, d is diameter of particle and D is diameter of container.

$$\frac{u_1}{u_2} = \left(\frac{1-c_1}{1-c_2}\right)^n \tag{18}$$

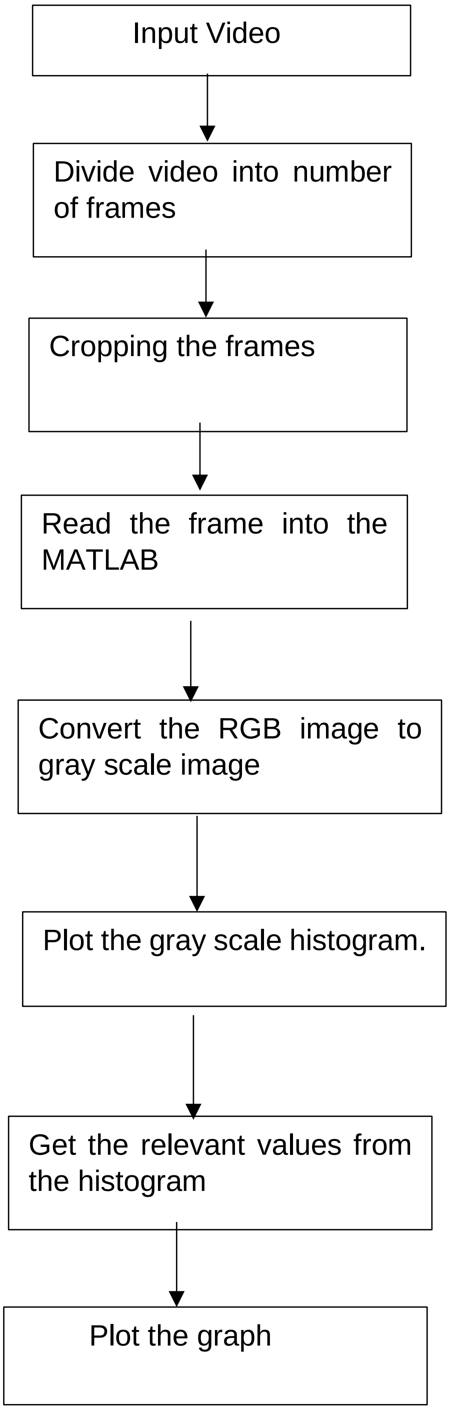
Where u_1 and u_2 are the values of sedimentation rates of suspension, c_1 and c_2 are the corresponding values of volume concentrations.

2.6 Sedimentation test for Arizona dusts

To limit possible inaccuracies caused by correction relations, the standard Arizona dust ISO 12103-1 was measured, among other things, for a volume concentration of (5 $\pm$ 0.5)%. The value was chosen regarding the validity of Zaki's relationship verified experimentally. These courses then form a graphic background, which enables a quick optical classification of the dust sample into categories A1 to A4 from the course of the sedimentation curve in the protocol. From the experimentally obtained values of representative particle diameter of Arizona dusts, intervals were created for the possibility of classifying dust samples based on the measured stokes diameter by sedimentation test.

As mentioned above, the course of the sedimentation curve in the height and time graph not only depend on the particle size but also depend on the concentration. This trend was confirmed experimentally on the reference test dust. As the concentration increase, the sedimentation rate of the suspension decreased, the course of the sedimentation curve is therefore flatter.

A video of the experiment that was conducted with the standard Arizona dust was taken, and this video is subject to image analysis in MATLAB. To perform the image analysis on the video below mentions things were done.



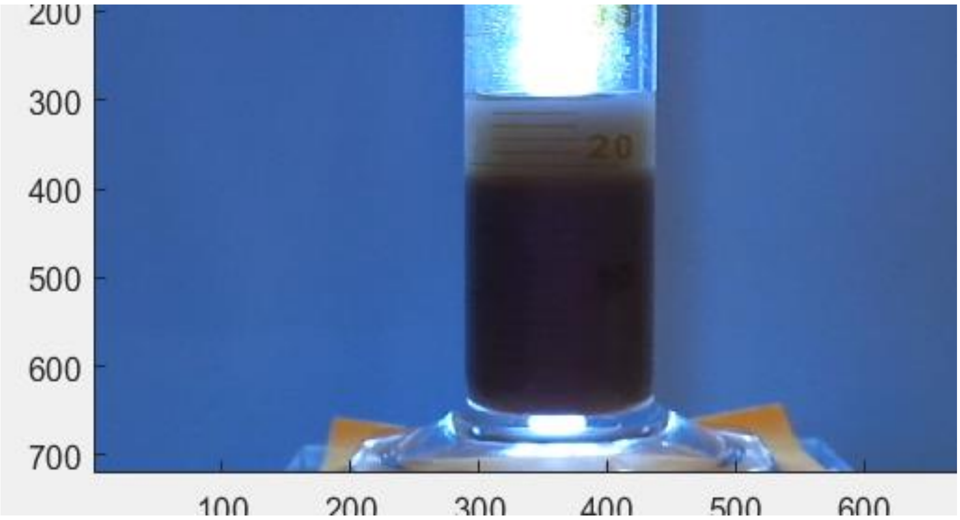


Figure 10: Image frame from the MATLAB

Figure 10 shows the image frame that was produced after the video was divided into the appropriate number of frames. We only need the area of the image where the sedimentation occurs in order to get the correct findings, hence we must crop the image.

Figure 11: Crop image frame

By using the function rgb2gray the image in figure 11 was converted to grayscale image.

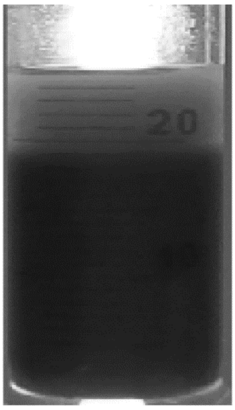


Figure12: Gray scale image

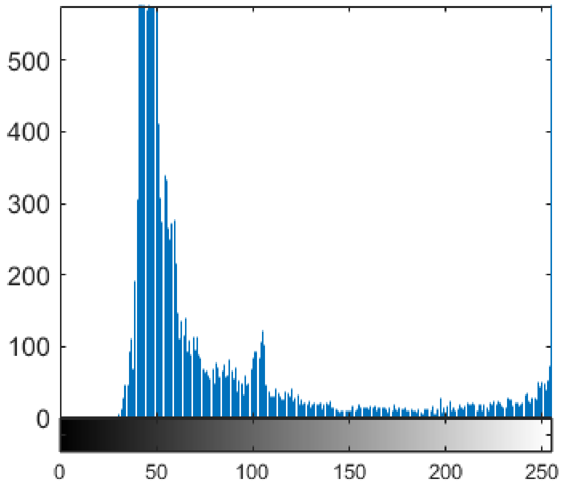


Figure 13: Gray scale histogram

Gray scale histogram shows the number of pixels in an image at each different intensity value found in an image. In figure 13 it can be seen that the intensity of sedimentation is higher, and the peak can be observed at ~40 and the value for example 500 as the time passes it can be seen that the intensity decreases.

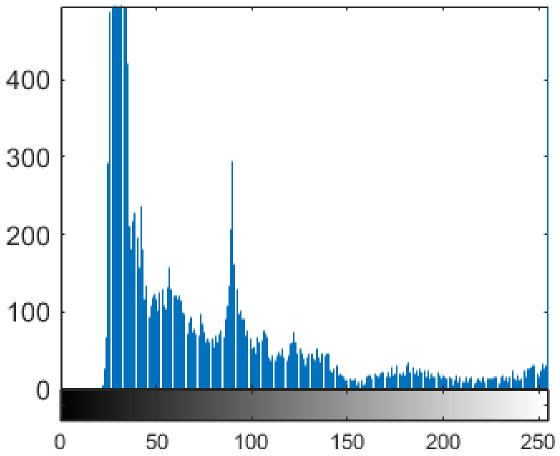


Figure 14: Gray scale histogram at the beginning of the Sedimentation

Figure 13 and figure 14 shows that there are different intensities at different time intervals during the sedimentation.

```
[counts,binLocations] = imhist(j);
[intesity,index1] = (max(counts(1:200,1)));
plot(0: (200-index1),counts(index1:200,1));
```

by using this function for every image, we can get the sedimentation curve from image frames.

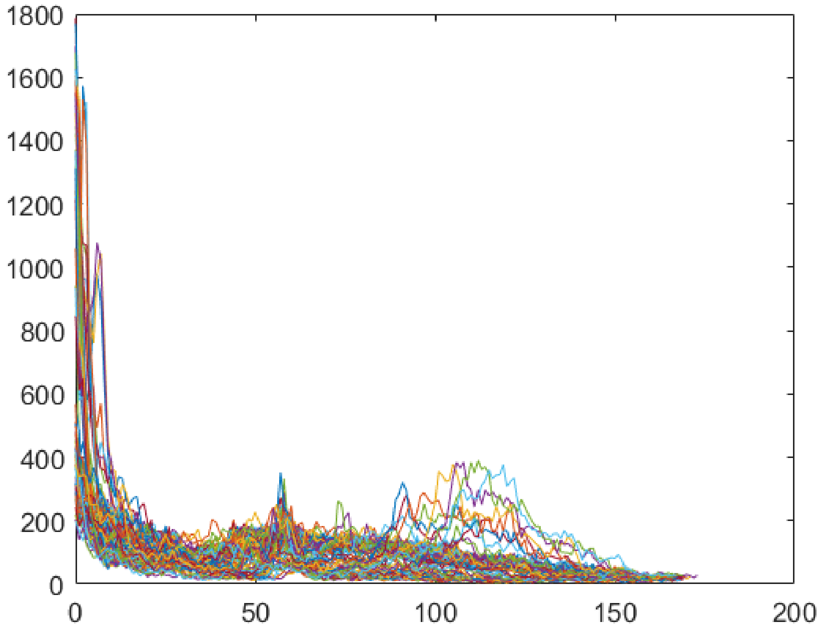


Figure 15: Intensity vs index curve from all image frames

Figure 15 is the collection of all 150 image frames graphs that were made from the sedimentation test video. Following this the x-axis was changed to time and the average of all graphs was calculated. Final graph is shown in figure16.

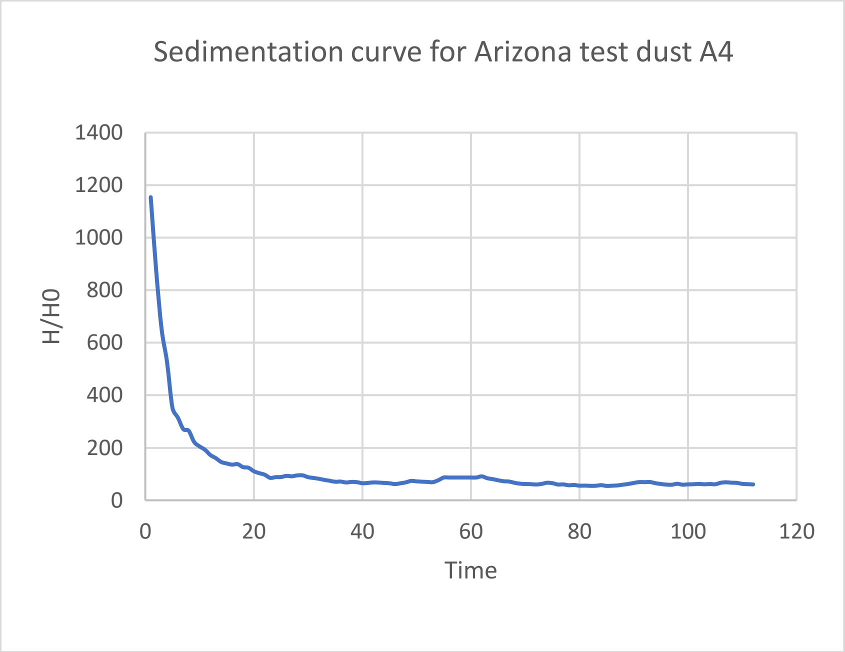


Figure16: Sedimentation curve obtained from automatic characterization of Arizona test dust A4

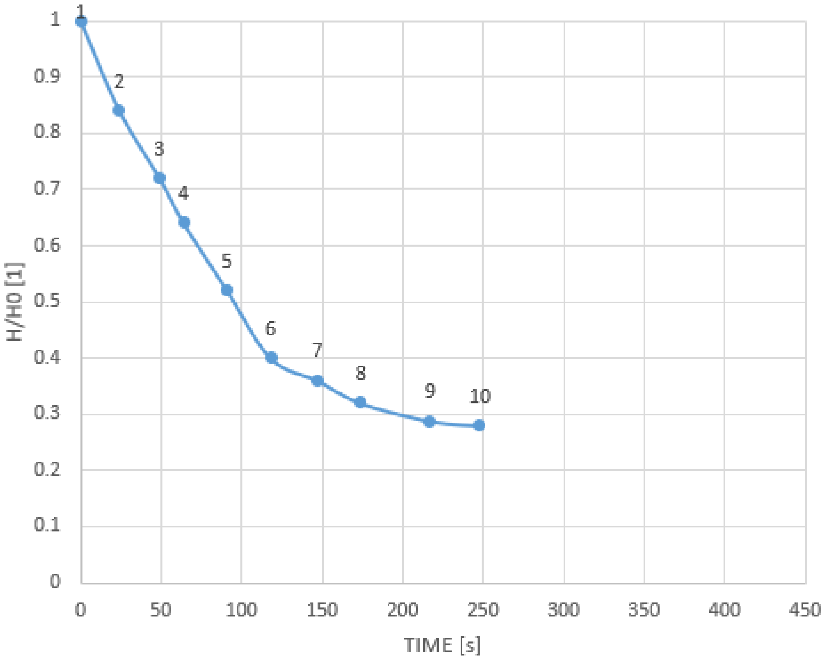


Figure17: Sedimentation curve for Arizona test dust A4

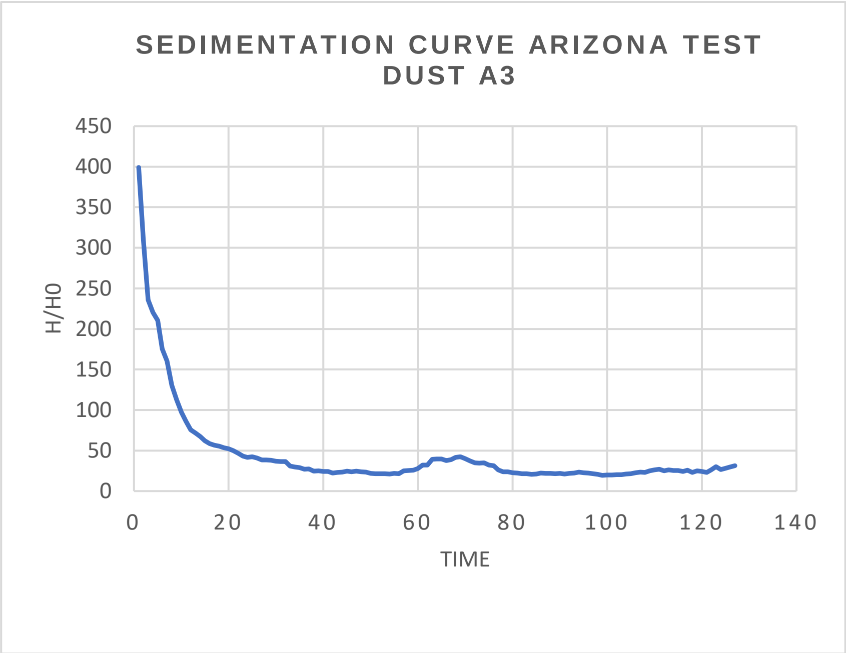


Figure 18: Sedimentation curve obtained from automatic characterization of Arizona test dust A3

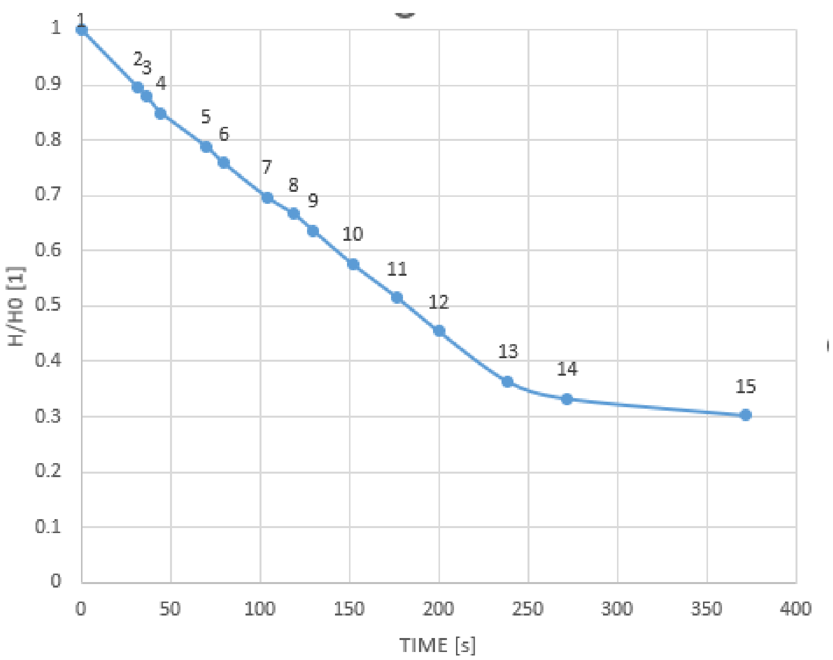


Figure 19: Sedimentation curve for Arizona test dust A3

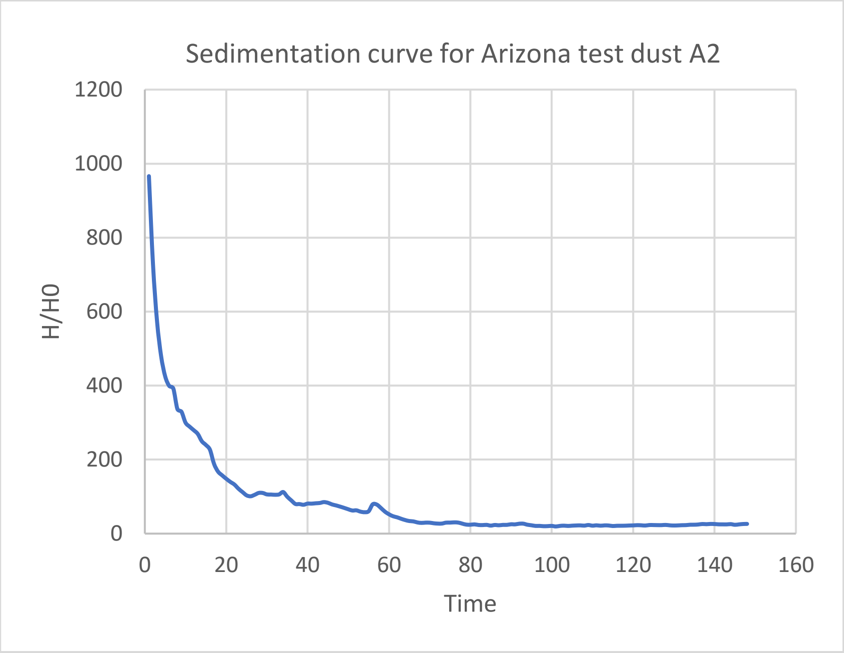


Figure 20: Sedimentation curve obtained from automatic characterization of Arizona test dust A2

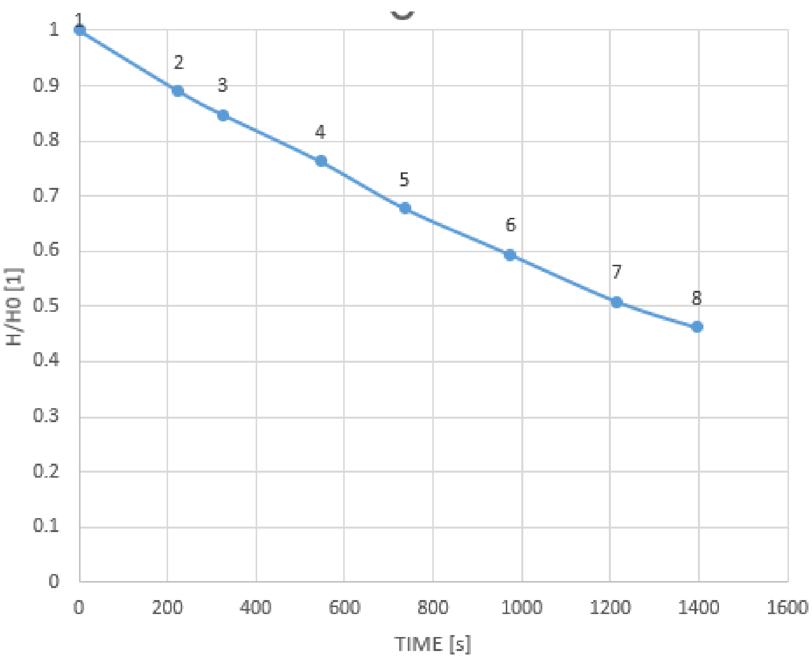


Figure 21: Sedimentation curve for Arizona test dust A2

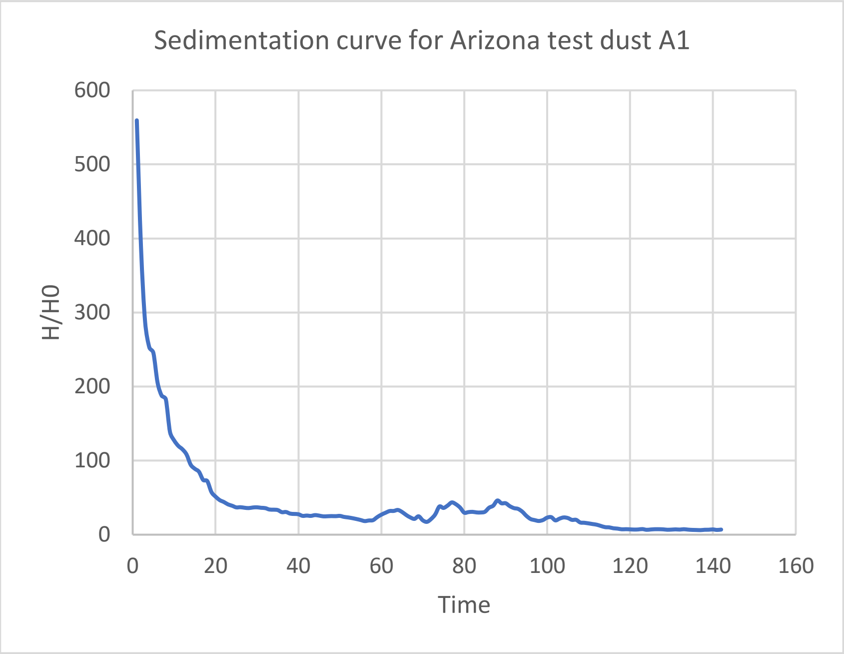


Figure 22: Sedimentation curve obtained from automatic characterization of Arizona test dust A1

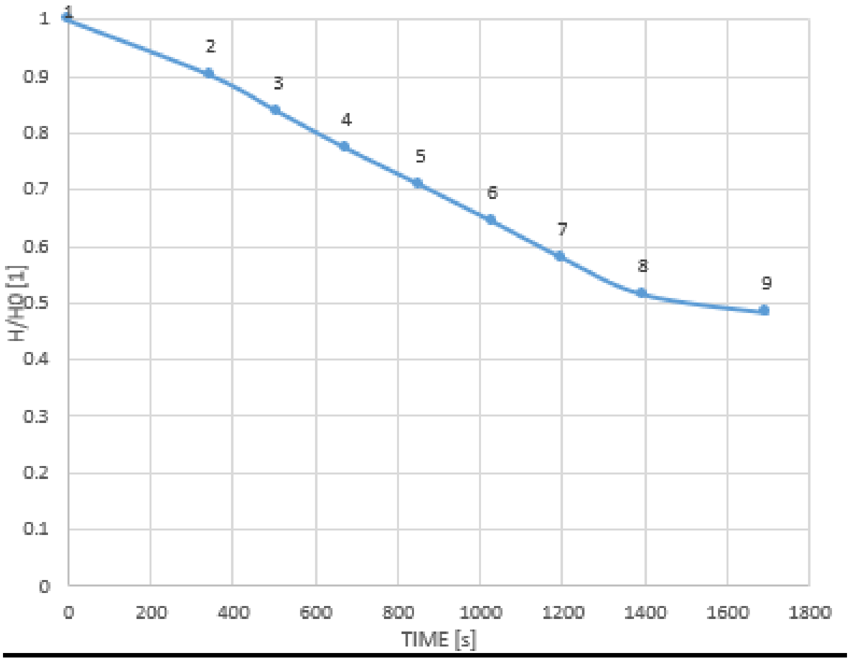


Figure 23: Sedimentation curve for Arizona test dust A1

The graphs in the figure 16, figure 18, figure 20, figure 22 were obtained after the image processing on the Image frames of the respective standard Arizona test dust.

The graphs in the figure 17, figure 19, figure 21, figure 23 were plot on the data that we gather during our experiment for respective standard Arizona test dust. With the help of this we also get the characteristic diameter and concentration for the respective Arizona dust which you can see in the table 3 below.

Dust sample	A1 Ultra-small	A2 Small	A3 Medium	A4 Coarse
Characteristic diameter [μm]	5.7	8.24	16.05	28.93
Interval for classification [μm]	< 6.9	6.9 - 12	12 – 17.2	>17.2
Concentration [%]	4.11	4.42	4.45	3.05

Table 3: Particles size values for classification into individual dust classes

2.7 Sedimentation test for Delivered samples

The sedimentation test of the delivered samples was performed according to the procedure describe in 2.3 sedimentation test procedure. The volume concentration interval for the legitimacy of the use of the Zaki relation was observed and considering the reference dusts, the samples were measured at a concentration of approximately 5%. A summary of the results is presented below (table 5).

Delivered samples	
1	5BK CEMA 19/2 RBTG
2	7BK CEMA 19/2 RBTG
3	5BK CEMA 19/2 IKA
4	7BK CEMA 19/2 IKA

Table 4: Designation of delivered samples

Sample	1	2	3	4
Sample weight [gm]	5.00	6.00	5.00	5.50
Volume fraction of sample in ethanol	2.55 %	5.92 %	5.82%	5.95 %
Respective particle diameter [µm]	18.61 µm	13.94 µm	13.41 µm	14.20 µm
Category of the Arizona dust	A4	A4	A3	A3

Table 5: Results of the sedimentation test of the delivered samples

2.8 Standardized procedure for classification of the road dust

The proposed method for the classification of road dust is based on the Sedimentation test. If the following measurement assumptions are met, the sedimentation tests measurements show good agreement in the classification of road dusts classification.

1. Use only dry cylinders.
2. For road dust, we recommend a graduated cylinder with a capacity of 50ml.
3. Weight an empty cylinder to form a balance. This value is used to check for possible cylinder contamination in the event of unusual results.
4. The distance between the lines on the cylinder is measured to convert the decrease in the level of the sedimenting particles from the volume to displacement, that is to measure the mark.
5. Weight 30ml of 96% ethanol into an auxiliary graduated cylinder and pour it from the funnel into the test graduated cylinder wetting the wall above the surface.
6. Weight the ethanol cylinder
7. The required amount of dust is scooped into a cylinder placed on the balance using a spoon – in the case of 30ml of ethanol, it is the range of 5gm to 10gm ideally 8gm of dust.
8. Homogenize the suspension with a homogenizer as gently as possible to suspend the particles.
9. At the end of homogenization, the timer starts
10. Level reading at time t=0
11. Deduction of interface height drop over time. Recommended reading after two millilitres (for a 50ml cylinder)
12. When the drop in the level stabilizes, the test ends
13. Values of the height of the interface and the time since the beginning of the measurement are entered in the protocol
14. The protocol evaluates the input data and proposes a dust classification into classes A1 to A4 based on the calculated particle size
15. Classification is also possible based on the comparison of the sedimentation curve with standards in the protocol. In case of disagreements, the primary determination is based on the particle size.

3 Conclusion

The content of this work was the collection of theoretical data for the analysis of the particulate systems to further measure sedimentation of concentrated suspensions. The knowledge gained from the literature was used in the experimental determination and evaluation of the sedimentation tests. Based on standard Arizona dusts ISO 12 103-1, the border values of the intervals for the classification of the supplied dust were determined. These values are summarised in the following (table 5). image analysis was done on the video of the sedimentation test And collaborates the results from the experimental data as well as the data from the image processing.

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Appendices

1. MATLAB Script for converting video into frames

```
% macro to extract frames and get frame means from movie
% and save individual frames to separate image files.
% Then rebuilds a new movie by recalling the saved
images from disk.
% Also computes the mean gray value of the color
channels
% And detects the difference between a frame and the
previous frame.
clc;    % Clear the command window.
close all;  % Close all figures (except those of
imtool.)
imtool close all;  % Close all imtool figures.
clear;  % Erase all existing variables.
workspace;  % Make sure the workspace panel is showing.
fontSize = 14;

% Open the movie
folder = fullfile('E:\Winter 2021-
22\Thesis\MP_ROOT\100ANV01');
movieFullFileName = fullfile(folder, 'MAH00138.mp4');

videoObject = VideoReader(movieFullFileName);
% Determine how many frames there are.
    numberOfFrames = videoObject.NumberOfFrames;
    vidHeight = videoObject.Height;
    vidWidth = videoObject.Width;

    numberOfFramesWritten = 0;
    % Prepare a figure to show the images in the upper
half of the screen.
    figure;
    % screenSize = get(0, 'ScreenSize');
    % Enlarge figure to full screen.
    set(gcf, 'units','normalized','outerposition',[0 0 1
1]);

    % Ask user if the individual frames shall be written
to disk.
    promptMessage = sprintf('Do you want to save the
individual frames out to individual disk files?');
```

```
    button = questdlg(promptMessage, 'Save individual frames?', 'Yes', 'No', 'Yes');
    if strcmp(button, 'Yes')
        writeToDisk = true;

        % Extract out the various parts of the filename.
        [folder, baseFileName, extentions] = fileparts(movieFullFileName);
        % Make up a subfolder for the movie frames.

        folder = pwd;   % Makes subfolder in the folder where m-file lives.
        outputFolder = sprintf('%s/Movie Frames from %s', folder, baseFileName);
        % Create the folder if it doesn't exist already.
        if ~exist(outputFolder, 'dir')
            mkdir(outputFolder);
        end
    else
        writeToDisk = false;
    end
    % Loop through the movie, writing all frames out.
    % Each frame will be in a separate file with unique name.
    meanGrayLevels = zeros(numberOfFrames, 1);
    meanRedLevels = zeros(numberOfFrames, 1);
    meanGreenLevels = zeros(numberOfFrames, 1);
    meanBlueLevels = zeros(numberOfFrames, 1);
    % spravne nastavit cetnost framu
    frame = round(logspace(log10(1),log10(numberOfFrames),150),0);
        for i = 1 : length(frame)-1
            if frame(i+1) <= frame(i)
                frame(i+1) = frame(i) + 1;
            end
        end
    frame
    for kon1 = 1 : length(frame)
        % frame = round(logspace(log10(1),log10(numberOfFrames),150),0)
        % frame = 1 : 100 : numberOfFrames
        % Extract the frame from the movie structure.
        a = frame(kon1);
        thisFrame = read(videoObject, a);

        % Display it
        hImage = subplot(2, 2, 3);
```

```
        thisFrame = thisFrame(:,300:end-50,:);
        image(thisFrame);
        caption = sprintf('Frame %4d of %d.', a,
numberOfFrames);
        title(caption, 'FontSize', fontSize);
        drawnow; % Force it to refresh the window.

        % Write the image array to the output file, if
requested.
        if writeToDisk
            % Construct an output image file name.
            outputBaseFileName = sprintf('Frame
%4.4d.png', a);
            outputFullFileName = fullfile(outputFolder,
outputBaseFileName);

            % Stamp the name and frame number onto the
image.
            % At this point it's just going into the
overlay,
            % not actually getting written into the
pixel values.
            text(5, 15, outputBaseFileName, 'FontSize',
20);

            % Extract the image with the text "burned
into" it.
            frameWithText = getframe(gca);
            % frameWithText.cdata is the image with the
text
            % actually written into the pixel values.
            % Write it out to disk.
            imwrite(frameWithText.cdata,
outputFullFileName, 'png');
        end

        % Calculate the mean gray level.
        grayImage = rgb2gray(thisFrame);
        meanGrayLevels(a) = mean(grayImage(:));

        % Calculate the mean R, G, and B levels.
        meanRedLevels(a) = mean(mean(thisFrame(:, :,
1)));
        meanGreenLevels(a) = mean(mean(thisFrame(:, :,
2)));
        meanBlueLevels(a) = mean(mean(thisFrame(:, :,
3)));
```

```
        % Plot the mean gray levels.
        hPlot = subplot(2, 2, 2);
        hold off;
        plot(meanGrayLevels, 'k-', 'LineWidth', 2);
        hold on;
        plot(meanRedLevels, 'r-');
        plot(meanGreenLevels, 'g-');
        plot(meanBlueLevels, 'b-');
        grid on;

        % Put title back because plot() erases the
existing title.
        title('Mean Gray Levels', 'FontSize', fontSize);
        if a == 1 %frame == 1
            xlabel('Frame Number');
            ylabel('Gray Level');
            % Get size data later for preallocation if
we read
            % the movie back in from disk.
            [rows, columns, numberOfColorChannels] =
size(thisFrame);
        end

        % Update user with the progress.  Display in the
command window.
        if writeToDisk
            progressIndication = sprintf('Wrote frame
%4d of %d.', a, numberOfFrames);
        else
            progressIndication = sprintf('Processed
frame %4d of %d.', a, numberOfFrames);
        end
        disp(progressIndication);
        % Increment frame count (should eventually =
numberOfFrames
        % unless an error happens).
        numberOfFramesWritten = numberOfFramesWritten +
1;

        % Now let's do the differencing
        alpha = 0.5;
        if a == 1 %frame == 1
            Background = thisFrame;
        else
            % Change background slightly at each frame
            %            Background(t+1)=(1-
alpha)*I+alpha*Background
```

```
            Background = (1-alpha)* thisFrame + alpha * 
Background;
        end
        % Display the changing/adapting background.
        subplot(2, 2, 3);
        imshow(Background);
        title('Adaptive Background', 'FontSize', 
fontSize);
        % Calculate a difference between this frame and 
the background.
        differenceImage = thisFrame - uint8(Background);
        % Threshold with Otsu method.
        grayImage = rgb2gray(differenceImage); % Convert 
to gray level
        thresholdLevel = graythresh(grayImage); % Get 
threshold.
        binaryImage = im2bw( grayImage, thresholdLevel); 
% Do the binarization
        % Plot the binary image.
        subplot(2, 2, 1);
        imshow(binaryImage);
        title('Binarized Difference Image', 'FontSize', 
fontSize);
    end

    % Alert user that we're done.
    if writeToDisk
        finishedMessage = sprintf('Done!  It wrote %d 
frames to folder\n"%s"', numberOfFramesWritten, 
outputFolder);
    else
        finishedMessage = sprintf('Done!  It processed 
%d frames of\n"%s"', numberOfFramesWritten, 
movieFullFileName);
    end
    disp(finishedMessage); % Write to command window.
    uiwait(msgbox(finishedMessage)); % Also pop up a 
message box.

    % Exit if they didn't write any individual frames 
out to disk.
    if ~writeToDisk
        return;
    end

    % Ask user if they want to read the individual 
frames from the disk,
```

```
    % that they just wrote out, back into a movie and
display it.
    promptMessage = sprintf('Do you want to recall the
individual frames\nback from disk into a movie?\n(This
will take several seconds.)');
    button = questdlg(promptMessage, 'Recall Movie?',
'Yes', 'No', 'Yes');
    if strcmp(button, 'No')
        return;
    end

    % Create a VideoWriter object to write the video out
to a new, different file.
    writerObj = VideoWriter('NewRhinos.avi');
    open(writerObj);

    % Read the frames back in from disk, and convert
them to a movie.
    % Preallocate recalledMovie, which will be an array
of structures.
    % First get a cell array with all the frames.
    allTheFrames = cell(numberOfFrames,1);
    allTheFrames(:) = {zeros(vidHeight, vidWidth, 3,
'uint8')};
    % Next get a cell array with all the colormaps.
    allTheColorMaps = cell(numberOfFrames,1);
    allTheColorMaps(:) = {zeros(256, 3)};
    % Now combine these to make the array of structures.
    recalledMovie = struct('cdata', allTheFrames,
'colormap', allTheColorMaps)
    for kon2 = 1 : length(frame)
        b = frame(kon2);
        % Construct an output image file name.
        outputBaseFileName = sprintf('Frame %4.4d.png',
b);
        outputFullFileName = fullfile(outputFolder,
outputBaseFileName);
        % Read the image in from disk.
        thisFrame = imread(outputFullFileName);
        % Convert the image into a "movie frame"
structure.
        recalledMovie(frame) = im2frame(thisFrame);
        % Write this frame out to a new video file.
        writeVideo(writerObj, thisFrame);
    end
    close(writerObj);
    % Get rid of old image and plot.
```

```
    delete(hImage);
    delete(hPlot);
    % Create new axes for our movie.
    subplot(1, 3, 2);
    axis off;  % Turn off axes numbers.
    title('Movie recalled from disk', 'FontSize',
fontSize);
    % Play the movie in the axes.
    movie(recalledMovie);
    % Note: if you want to display graphics or text in
the overlay
    % as the movie plays back then you need to do it
like I did at first
    % (at the top of this file where you extract and
imshow a frame at a time.)
    msgbox('Done');
```

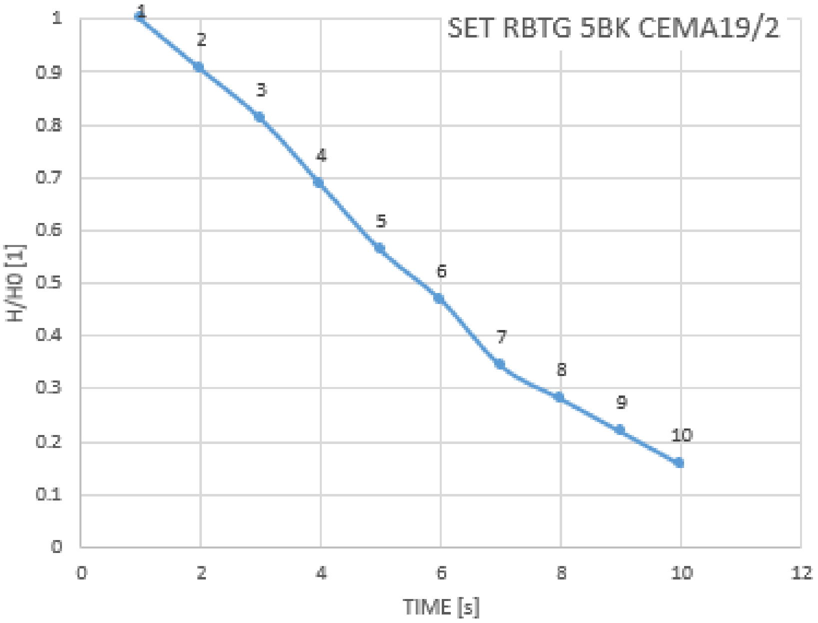

Figure24: Sedimentation curve for delivered sample 5BK RBTG CEMA 19/2

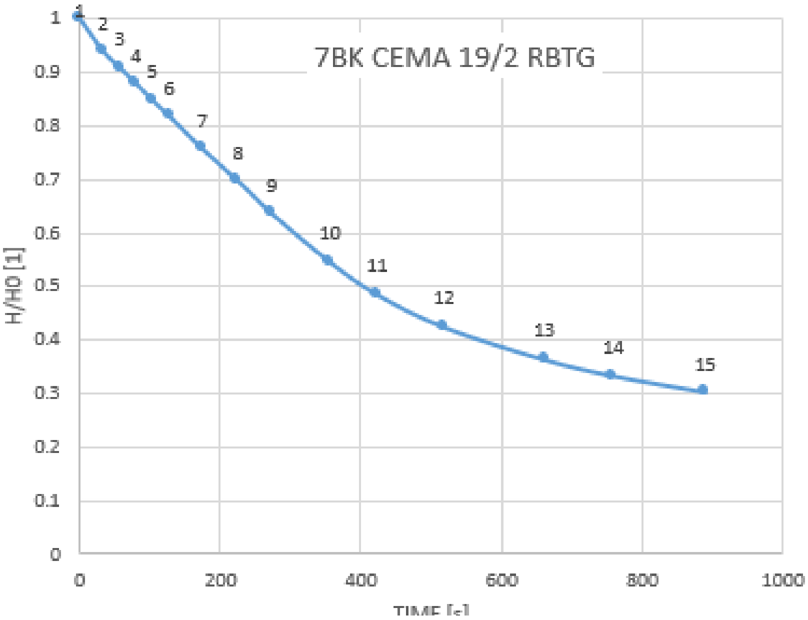

Figure 25: Sedimentation curve for delivered sample 7Bk CEMA 19/2 RBTG

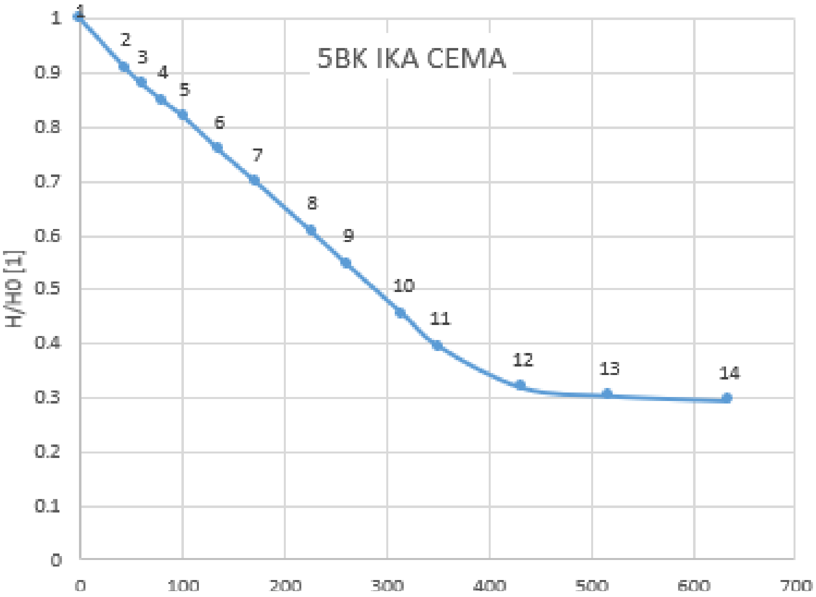

Figure 26: Sedimentation curve for delivered sample 5BK CEMA 19/2 IKA

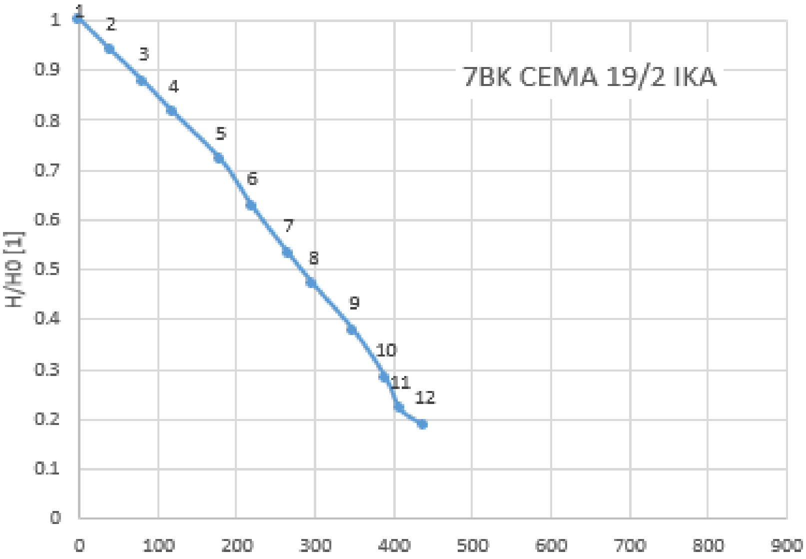

Figure 27: Sedimentation curve for delivered sample 7BK CEMA 19/2 IKA