Aggregation of Particles in a Rotating Magnetic Field

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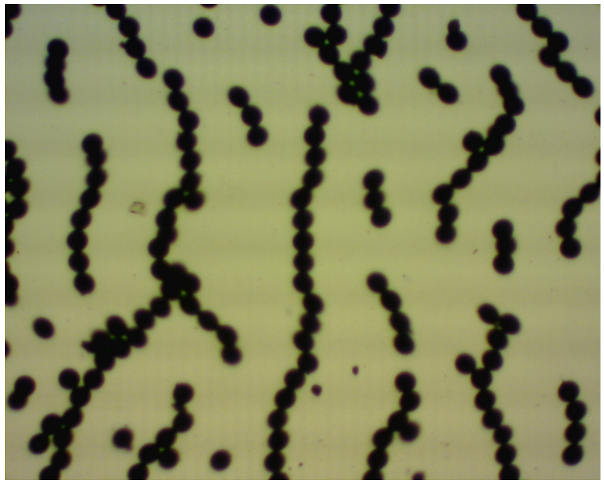


Figure 1: Magnetic particles in a linear magnetic field.

Aggregations of Magnetic Particles in a Magnetic Field

This rapport is the evaluation criteria of the course "FYS3180 - Eksperimentelle metoder i fysikk", experimental methods in physics, at the University in Oslo, autumn 2013.

My project assigment was "Aggregation of Particles in Fluid by use of a Rotating Magnetic Field". My supervisor was Geir Helgesen, Institute of Energy Technology (IFE), Kjeller. The assignment was executed in at the physics department at the IFE, Instituttveien 18, Kjeller.

Project description

The physics group at IFE have been developing magnet technology for the use of seperation of micro and nano particles. Here the particles are used to filter out cells and microbes in the blood. The particles would be treated with an agent that sticks a target substance, for example cancerous cells, to the particles and with a magnetic field the particles would be pulled out of the solution for further analysis. But as of yet this is not a very effective method to seperate the particles out of the solution, as it takes a long time and a very strong magnet is needed. The challenge would be to find a good method to remove particles from the liquid.

The goal of this project is to instead of using a static magnetic field, use a oscillating magnetic field to try to gather particles in bigger aggreagtions. The particles used in this project are magnetic particles made out of polysterene with iron oxide $4.5\,\mu$m and we want to look at the properties of these particles and see how well they aggreagate. The iron loaded particles are made in the the Ugelsta labaratory at NTNU/SINTEF. With this method we ensure that the spheres are very similar to each other. Non magnetic particles in a ferro fluid are also used to compare the results gathered from the iron loaded particles. Digital picture handling is used to analyse the results under a microsope.

Figure 1, shows a prior experement done at IFE where particle aggregation were shown in a linear static magnetic field. In figure 2 and figure 3 we can see the iron loaded particles and the non magnetic particles in a ferro fluid used in this project.

Introduction

Aggregation of particles is a common phenomena in nature and is also exploited in many manufactoring uses, such as papermaking and cheese making, by the use of a coagulating agent. The phenomena of aggregation can be described as particles gather around each other in clusters and often forming complex patterns. Crystal formation can also be viewed as a aggregation phenomena.

In this article both magnetic iron loaded particles and what is called magnetic holes is used. The term magnetic holes was described by a research group at IFE, where my supervisor was a part of. The term

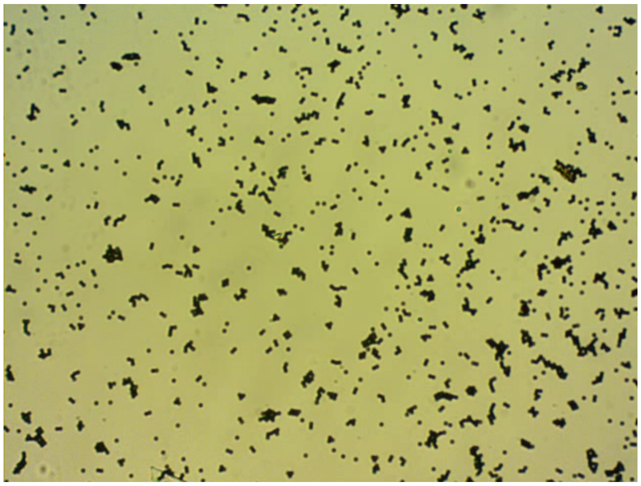


Figure 2: Magnetic particles in a oscillating magnetic field.

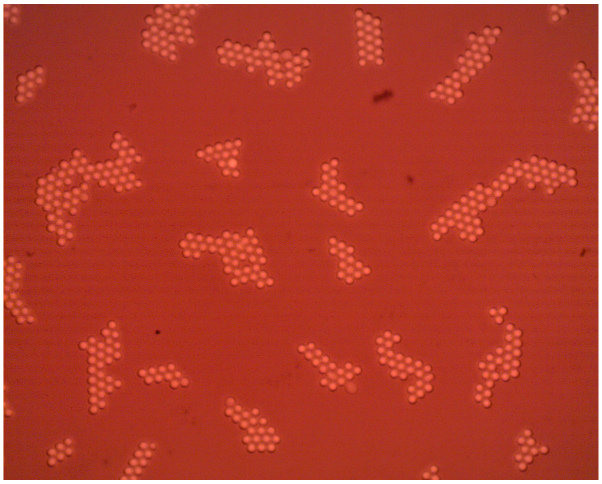


Figure 3: Non magnetic particles in a ferro fluid in a oscillating magnetic field.

besically describes non magnetic particles in a magnetic fluid. It was descovered that these non magnetic particles would start to act like magnetic particles because of the magnetic fluid around them. With their procedures they were able to get the magnetic holes to gather very smoothly, without sticking to the glass of the sample, and they were also able to make the particle move away from each other by applying a magnetic field, normal to the sample.

In this project we would like to look at magnetic particles of iron and see how well they aggregate. If we found magnetic particles that have good aggregational properties it means that these particles might be valuable for further research. And could then also have different application areas, such as in medicin to filter out spesific cells or microbes.

Aggregation Theory

The main goal of this project is to use a oscillating magnetic field to get magnetic particles to gather. To do this we have used a series of coils to create a magnetic field which oscillates in the x and y direction of the plane. The particles becomes magnetic when they are exposed to the magnetic field and because they are sufficiently small they can be treated as magnetic dipoles. In the case of the magnetic holes, the non-magnetic particles would be given an efficient magnetic dipole moment from the magnetic fluid around it. Thus they act like normal magnetic dipoles.

Actually calculating the force between each dipole is quite complicated, but it is sufficient to say that the force between each dipole can be given by a function which is proportional to a trigonometric function cos or sin divided by a distance relation,

$$F \propto \frac{\cos(\theta)}{r^x}.$$

By oscillating the magnetic field we ensure we get a sum attraction of the particles.

With ideal particles, the clusters would gather into bigger and bigger aggregations with time until there was only one big cluster in the middle of the sample. The clusters would first gather with single particles into clusters of pairs and then continue gathering into exponentialy bigger clusters until there is no more clusters left. My supervisor has priorly worked on aggregational problems and argues that the size of clusters can by given by a function $S(t) \sim t^z$[1] (we see that this kind of function fits the data very well). By plotting the results in a log vs. log plot we can get the constant z as the slope of the function.

$$\begin{aligned} S(t) &= t^z \\ \log S(t) &= z \log t \\ x &= \log t \\ \log S(t) &= z \cdot x \end{aligned}$$

A discrepancy from this behavior may suggest that there are other factors in our experement that influence the aggregation pattern. Because we have conservation of particles, the increase of the size of the clusters means that the number of clusters must decreases at the same rate. We then have a function for the total number of clusters, defined as $N(t) \sim t^{-z}$.

We define these two functions as,

$$\begin{aligned} N(t) &= \sum_s n_s(t) \\ S(t) &= \frac{\sum_s n_s(t)\, s^2}{\sum_s n_s(t)\, s}. \end{aligned}$$

Where $n_s(t)$ is the number of clusters of size s at time t, s is the cluster size and $S(t)$ is the weighted mean of the cluster size. Here the weights are chosen as $n_s(t)\, s$, which is the total number of particles in clusters of size s.

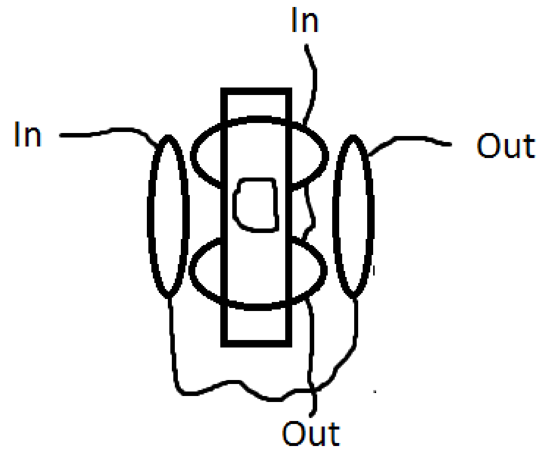


Figure 4: Simple schematic of how the sample area was surrounded by coils. There was also set up at coil above and beneath the area.

Experiment

In the experiment we use a optical microscope to view the sample. A camera is connected to the microscope and feeds the images to a personal computer we had on site. The program used to capture the images was Dino-Lite pro. The samples was created by my supervisor, Geir Helgesen, who made the samples using the iron loaded particles, and the particles used in ferro fluid sample. The particles lay between two glass plates which was seperated by the use of some bigger particles than the particles we wanted to analyze. These bigger particles gives the iron loaded polystyrene microspheres and nonmagnetic microspheres mixed in ferrofluid room to move more easly in the sample, but also in some cases was the reason we could not use certain areas for analyzing because they would ruin the image area. The two glass panes on either side of the particles were glued to each other.

To create the magnetic field, three pairs of coils were set up around the sample. Figure 4 is a basic scetch of the sample area. The ellipses represent the coils. There was also a coil above and beneath the sample area so we would be able to apply a normal magnetic field if we wanted too. The measured magnetic field strength was 795 A/ m, a field about 20x the earths magnetic field. The rotation frequency of the field was set to 300 Hz.

Priorly my supervisor had made the observation that some times that particles would stick to glass and was unable to move well. A suggested reason for this was that the glass often has a surface charge and that the particles also often has a surface charge that makes them stick. Because of this all the glass slides were treated with a substance Lecithin that would make a neutrually charged layer between the glass and the sample. This would hopefully reduce the columb force between the glass and the particles.

First we conducted experiments with the samples with the magnetic holes. The week after the inital experiments we conducted the same experiment with the iron loaded particles. In all 8 samples of iron loaded particles was made while only 4 of them we were able to use in the experiment. There was made 2 samples with the particles in the ferro fluid and both of them we could use in our sample. The iron particle samples were used two days after they were created. One week after the experiments with the iron loaded particles none of the samples with iron loaded particles could be used, either most of the particles would stick to the glass or some of the water would have evaported. This suggests that not all our samples were sealed properly.

To prepair the sample we first used the optical microscope to see check the distribution and if the particles were too close to eachother we tried to apply a normal magnetic field on the magnetic holes (which had been tested before). Another method that was used, was to put the sample in a ultra sonic bath. This was a simple small tub that would shake the sample at frequencies using ultrasound in order to spread the particles more appart.

In the ferro fluid the non-magnetic particles had the size $14\,\mu$m, the iron loaded particles had a size $4.5\,\mu$m. As a side note one can see that from some of the magnetic hole images two and two particles are stuck together from the start. This is because we seperated the particles from each other by setting up a normal field through the sample, this makes them seperate in the xy plane but makes them stand on top of each other in the z plane. When they relax they 2 and 2 next to each other because that was the hight they were able to grow with the normal field.

Numerical Methods

All programs and results can be found on GitHub at the url

https://github.com/lastis/ParticleStorm

. Note that the movies and pictures used for analyzation is not found here because of they take up a size above 1 Gb.

All our results are found by using numerical methods and digital picture handeling to analyze the pictures. This was done using a library callen OpenCV, used with the Python interface. The reason for chosing this library was because it is programmed in C++ and is thus a very fast library. It is also a open source project and has good reviews of its perfomance and efficiency. OpenCV's primary interface is C++ but also has many other interfaces, such as Java, Python or Matlab. For this project the programming language used was python because of its ease of use and simple array handling.

OpenCV had many paramters that had to be set to be installed correctly, so a shell script was used to (on Ubuntu) to install the software. The version of OpenCV used was v2.4.6.1 and the shell script used can also be found on github.

Image Handeling

To analyze the pictures a number of methods were used to make the pictures become black and white where the particles would be shown as white. All images had timestamps on them and these were used to rename the images into seconds so a timeline could be made. To analyze clusters, particles next to eachother would be connected by first using a blurring to a thershold to make clusters whole areas of white. In this way the clusters would be shown by complete white and the single particles inside of it could no longer be visible in the treated image. This was done to make it more simple to analyze the image using image contouring.

To make the movies into images a library called ffmpeg was used. This library is now deprecated, but still serves our purpose well. The command line was:

```
 ffmpeg -i MOVIENAME.FILTYPE -r
0.05 -f image2 image%3d.bmp
```

The the input value after -r is $\frac{1}{\text{frame rate}}$, so $0.05 = \frac{1}{20}$, which gives us one picture every 20 seconds.

The thresholding was executed by first blurring the image to get rid of noise in the image and make particles close to each other blend. After that, Otsu's thresholding method was used to seperate the particles from the surroundings. Otsu's thersholding method automatically finds a good threshold value by assuming two classes of pixels defined by the image histogram of the pictures.

In some cases a adaptive thresholding algorithm had to be used because the background of the image had different lighting in different parts of the image. In these cases the adaptive method was used first and then Otsu's method was used afterwards to get a good result.

After the thresholding was done, the pictures became only black and white images where the clusters would be shown in white. See picture figure (5) for the untreated image and figure (6) for the treated image. At this point image contouring was used to count the number of clusters and then also the cluster size (in measure of pixels). By taking the cluster size and dividing by the area of particle the final cluster size was found (in number of particles). If clusters would have areas without particles inside of them, this area would be subtracted. This way of treating the images is simple but because the particles are blurred into each other it is also a source of errors for our results. In our sample data there was a difference between the

Figure 5: Original picture of particles in the ferro fluid. The highlighting in the middle of the picture ment an adaptive thresholding had to be used.

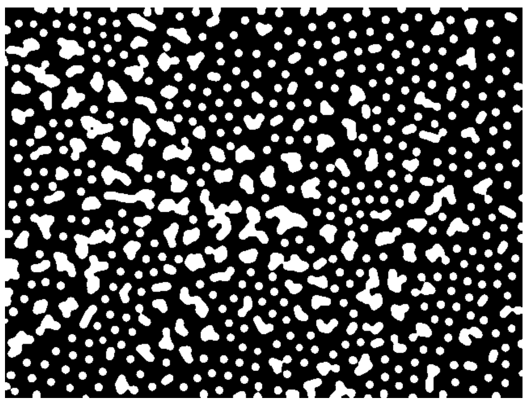


Figure 6: Same image as figure (5), but after blurring and thresholding.

images captured by the camera mode and the picture mode of the image software. The camera mode had lower resolution and also had a smaller field of view. Because of this we had to allow two sets of particle sizes (in pixels) for one set of measurements.

In the program the clusters were represented as arrays of all the contours in the image. The length of this array is $N(t)$, which is the numbers of clusters in the image. $S(t)$ was computed by taking the weighted mean of the area of clusters (contours in the program). The last thing to be calculated was the linear regression of the data. This was done using the NumPy library for python with the function linregress. The values from this regression was used to recreate the red line that can be seen in the plots. Both the slope and the R value is printed to the plot.

To find the size of a particle the methods described above were used on a calibration image (an chosen image in a set of measurements). Then a spesific cluster would be chosen and the particles in that cluster would be counted manually. That number were put into the program and the particle size (in pixels) would be printed to the terminal screen.

Description of the programs used in the project (can be found on github):

ProcessMethods.py : A collection of methods used in the other programs.

CalculateParticleSize.py : Program used to test thresholds and find the particle size needed in ProcessImages.py

PrepairImages.py : Program used to apply threshold on all the pictures in a given folder.

ProcessImages.py : Gets the area of all the clusters as an array and saves it to a txt file.

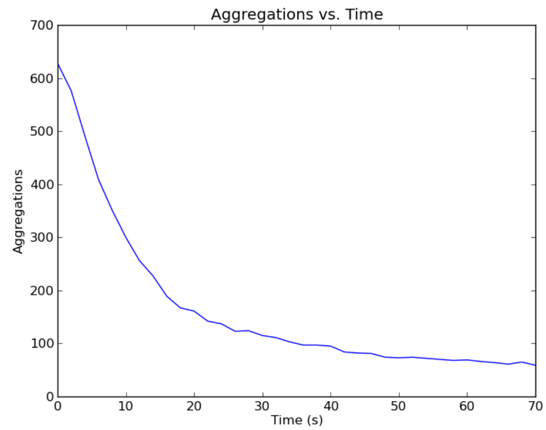


Figure 7: Fast aggregation of magnetic holes.

PlotParticles.py : The program that calculates $S(t)$, $N(t)$, finds the linear regression and creates the desired plots.

Sources of Errors

During the experiments we discovered that some of the samples had evaporated the week after they were created. This ment that those samples could no longer be used. Also some of the iron particle samples were ruined because pockets of air appeared after they were put in the super sonic bath.

The way the particle size was calculated gives rise to error because the particles are blurred into each other to make clusters whole areas of white. In this way the cluster size is always bigger then the actual area and this makes bigger clusters contain more particles than the particles that made them. To try to negate this the program counts the number of particles in the first image used for a set of images. It then multiplies all cluster sizes with a factor to ensure that the same number of particles are contained in each picture.

The be able to find good areas of the sample to analyze we had to find areas where the particles had a good enough distribution to fit in the camera's view and where clusters had not already been formed. Because of this the center is often outside of the camera view and there is a flux of particles and clusters entering and exiting the camera view. This trend can be seen in some of the pictures.

The program used to capture the images, Dino-Lite Pro, had a slightly different area of view from the camera mode to the picture mode. This affected out results and the program had to be modified to allow two sets of particle sizes in one set of measurements.

Discussion

The non-magnetic particles in the ferro fluid had a much smoother and faster aggregational pattern than the iron loaded particles. This was both confirmed in the way they aggregated together and the way the seperated when a normal field was applied to the sample to make them seperate from each other so we could make new measurements with the same sample. The particles in the ferro fluid were compared to the data from the iron loaded particles. An example of the fast gathering of particles can be shown in figure 7. We notice that the amount of clusters goes to one sixth of the start over the course of 70 seconds.

It was discovered that the iron loaded particles stuck to the surface of the glass very easily. In most cases the some of the iron loaded particles would stand still during the whole measurement. The result was that when new particles clustered together with these particles, the newly formed cluster would now only spin around the hook point of this stuck particle. It seems that was the reason many of samples could not be used.

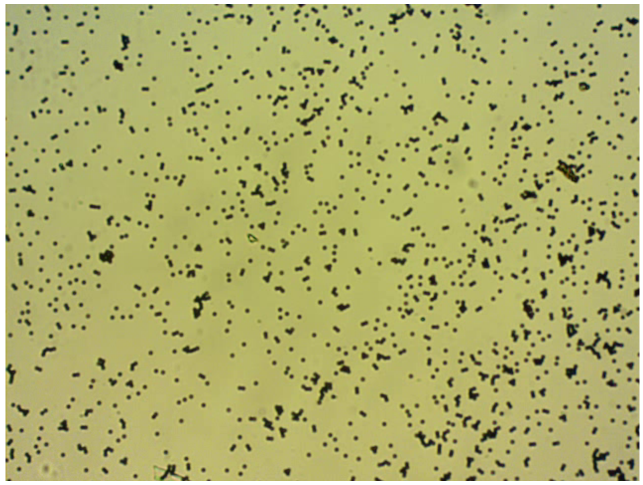


Figure 8: The first state of our best measurement on iron particles.

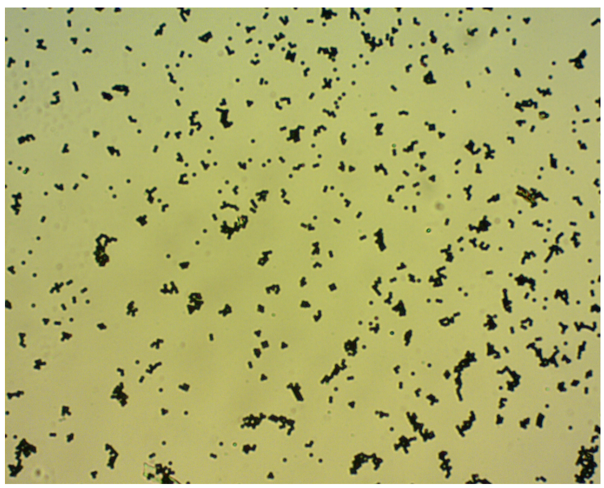
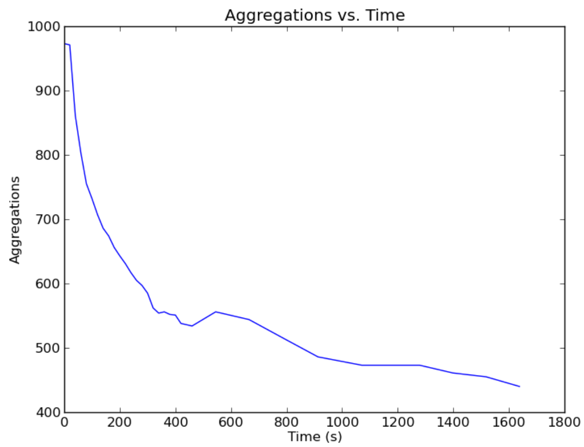


Figure 9: The final state of our best measurement on iron particles.

From the theory have that the exponent z in the size of the aggregations, $S(t)$, should be opposite of the z value of aggregations $N(t)$. We discovered from the images that the number of aggregations was a much more smooth graph than the particle size $S(t)$, and would suggest that counting aggregations is a better indication of the change in system then the size of the aggregations. The reason for this is assumed to be because of the inaccuracies in the algorithm used to find the size of the clusters. The counting of clusters was a much easier and apparently more accurate way to do this analyzation. In figure 10 we see the results from our best measurement with iron loaded particles. The initial and final state of this system can be seen in figure and 9.

With these plots we did a transformation to a log vs. log plot to find if the particles fit the curve given in the theory. For the plots with the most smoothest aggregation pattern the slopes seems to fit very well. But this was not the case of all of them. The plots from all the measurements from the magnetic holes are given in figure 11 and figure 12. The last one of these plots arguably is the best one.

The slopes from the iron loaded particles are given in figure 13, and 14. In the case of the second measurement here, figure 14, there was a greater flux of particles leaving the image than in other cases. The reason for this is probably because we are far from the center of the sample and the magnetic field did



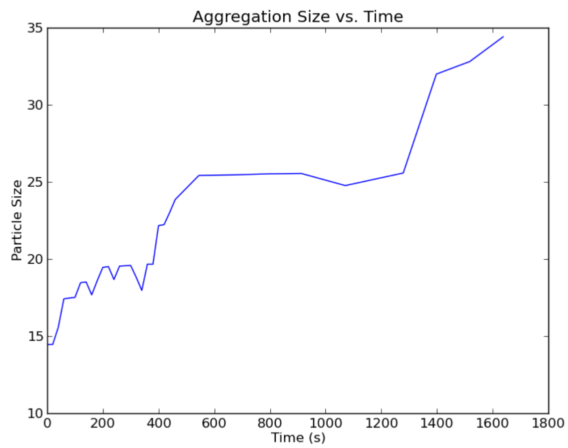


Figure 10: These are the plots from the best measurement of iron particles. Note the smoother curve in the top plot.

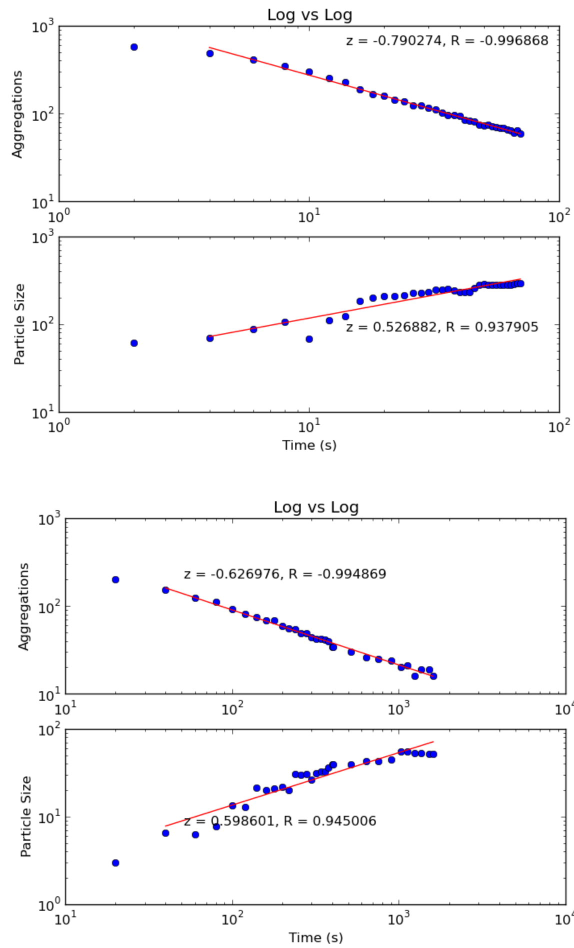


Figure 11: Results from 1. and 2. measurement with magnetic holes.

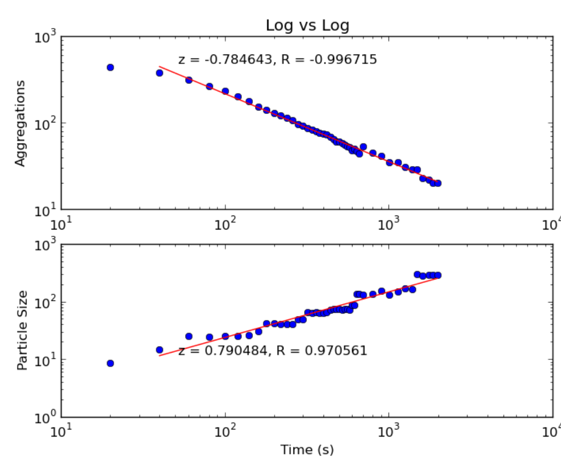


Figure 12: The results from the 3. measurement with magnetic holes.

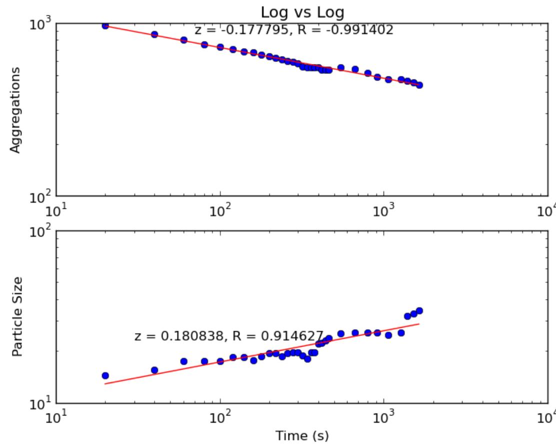


Figure 13: Results from the first measure with iron particles.

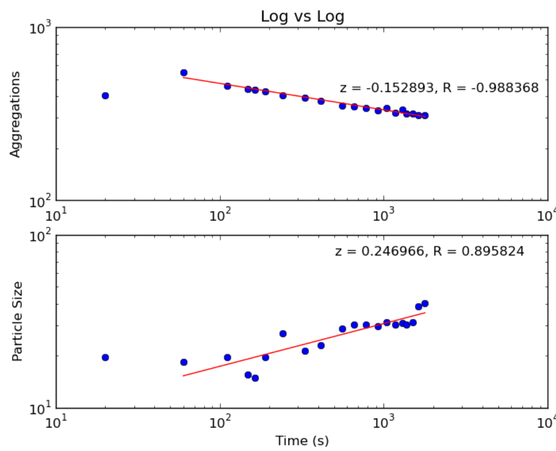


Figure 14: Results from the second measurement with iron particles.

not oscillate circulary. We had 4 measurements of the iron loaded particles but two of them was discarded as most of the particles never moved and an aggregational pattern was not observed. In the other two measurements the iron loaded particles did not seem to stick to the glass as much. The first plot belongs to the pictures in figure 8 and figure 9. The two slopes of the first measurement of iron loaded particles is very close to each other. This fits with the theory we described before. The value of these are $z = -0.177795$ and $z = 0.180838$. It can also be noted that the R values of these two slopes are better then for the second measurement of iron loaded particles.

The reason why the slopes of second measurement of iron loaded particles does not fit each other well is thought to be bacause of many clusters leaving and entering the image, that the polarization of the field is not very circular at this point and that particles tend to stick to the surface.

In the two iron particle measurements, there was a tendancy for bigger aggregations to stop moving after they connected to particles that were stuck. It therefor seems that the force from the bigger aggregations were not strong enought to pull loose the stuck particles. In some cases we could see that bigger aggregations rotated around spesific particles and not their apparent center of mass. This further suggests that some particles could make the aggregational pattern stop for a whole cluster.

We also discovered that when using the super sonic bath to shake to sample in order to change the particle distribution, for the iron loaded particles bubbles would form underneath the glass. This was surprising and made those samples unfit for analyzation. This might be because the particles are made of a material that is porous and contains pockets of air. Although we were not sure of this and it could also come from some different contaminant, like a hole in glue on the side of the sample. This happend for two of our samples.

Conclusion

We were able to see that cluster formation was present in most of our experements, and all of the experements done with magnetic holes. We further showed that they seems to tend to a function t^z where the z value could be gathered from analyzation of the data. When the same methods were tried applied to the iron loaded particles, not very good results were gathered. Most of our samples could not be used because of their tendency to stick to the surface of the glass on the samples. But in the cases where we were able to do measurements there seems to be the same aggregational pattern. The aggregational patter of iron loaded particles was much slower than for the magnetic holes. This can be seen from the from the z value in the plots, for the non-magnetic spheres we have a z value which ranges from 0.52 to 0.79, for the iron loaded polysterene particles we have a $z \sim 0.2$.

We also observed that small particles that stuck to the surface of the glass would stop bigger clusters stop moving. In some cases the bigger clusters would just spin around the particle that was stuck on the glass. Because of this we see that freedom of movement is a crucial element for cluster formation.

Assuming the theory holds and the aggregational size and number can be given by $S(t)$ and $N(t)$, we also found that counting number of aggregates was a more accurate way of analyzing the data on the computer. Thought this is a conclusion that is spesific for our method of analyzing the pictures. If the cluster size measurement method were more accurat this might not have been the case.

Possible future improvements

Most of our iron particle samples could not be used, here there is definitely more research that can be done to make better samples. An increase of measurements of both magnetic and iron loaded particles would also yield more definite results as maybe a more definite aggregational patterns could be found. This project also had time constraints as it had to fit in a Norwegian school semester.

If the particles had better ability to move maybe the force on the different aggregations could be measured. To find out if bigger aggregations could actually be pulled out or concentrated in a spesific area of the sample. In this way maybe the particles could be used for further reasearch in amongst medical applications. It will be exciting to see further reaseach in this field will yield results.

Acknowledgements

I would like to greatly thank my supervisor, Geir Helgesen, for his hospitality and his work for preparing the reasearch area so the experements could be conducted. I am also very appreciative of IFE and their willingness to work with the University in Oslo to take in students for them to work on a project like this.

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