Why size matters

How fraction size influences the soluble nutrient composition in an extract of ground hay and ground characteristics

Entry to the Stockholm Junior Water Prize 2021
'Why size matters'
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Summary

Soluble content is relevant for the research for fertilizers because they have a big influence on the way and efficiency plants grow. Shortage of certain nutrients can sometimes withhold a plant from growing or performing certain processes. Also too much of some nutrients can damage certain processes of the plant and can even lead to the death of the plant. Which answers the two subquestions.

Not only is soluble content and particle size in land relevant for plant growth and survival, it also influences the economy together with several other factors. When soil degrades through several different factors, the crop yield of the grounds also decrease which influences the amount of crops the farmer can sell, which gives a decrease of supply with the same or growing demand. That increases the price of food, even in area's where food is scarce. This means the world pays for degrading soils and is also a reason why protecting soils is very important.

This report shows the relation between particle size and the forthcoming soluble content. I show this by an experiment with ground hay in water. When different sizes of ground hay is added to water, a variation of colour can be noticed. When the particles of hay are fairly big, the colour of the water will turn slightly brown. When the particle size decreases, the browning of the water increases. This indicates a higher value of soluble content of the hay. This gives us an answer to the research question: 'In what way does soluble fraction of nutrients change after size reduction?' The hypothesis is true in most cases with the nutrients. Although not every soluble nutrient content of every ion or acid increases with smaller particles, normally it does. I think this has to do with the fact that the experiment was relatively small. The second experiment shows the principle of faster absorption the bigger the particle size is in the top layer. It shows the denser the mixture of the soil, the better it retains water but the slower it absorbs water.

The first experiment consisted of a few steps. Firstly I ground the hay in several different sized fractions after which I weighed them and put it in water. After mixing and centrifuging the samples I made dilutions and made smaller samples out of them which could be put in several different analyzers. After the samples were analyzed, I got all of the results.

For the results of the second experiment a thorough analysis was not needed, because of the vast amount of variables in the situation in which the technique can be implemented.

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Introduction

As the world's population grows, the demand for food is growing as well. All of earth's mouths have to be fed. Which should not be a problem, if we all handle our agricultural ground with all love and care it needs. But more has to be done if we want to feed all estimated 9.7 billion people on earth by 2050. Which brings us to a possible solution for the growing problem of getting too little crops from the existing agricultural acres of lands. Fertilizer in various forms have been used throughout the years with great success, but some sorts have a negative side to them.

Hay or other dried or burnt plants are often used as a eco-friendly way of fertilizing and improving the characteristics of land. As the plants still contain the organic content from when it was still alive. That brought me to the subject for this project and also the research questions:

'In what way does soluble fraction of nutrients change after size reduction?'

And:

'How does an addition of dead organic material to soil influence the characteristics of the soil?'

Of which I got the subquestions:

'Why is the soluble content relevant in research on fertilizers?'

And:

'How do cations and anions influence the growth of plants and the efficiency of agricultural soils?'

I expect that the smaller particles will have a higher soluble content. Just like catalytic converters work better when they are made into smaller particles. In that case every particle will dissolve easier than the same kind of nutrient, but in a bigger particle. Secondly, I expect that the soils will absorb water faster the bigger the added particle size is, but will hold water worse than a more dense composition.

The method of gaining the results for the answer to the research questions was a very straightforward one. With the help and collaboration of Wetsus Research Institution and the use of their laboratory I got the results needed. I will discuss the detailed method and materials later on in the report.

This report will consist of a literature review, followed by the results, answers to the research question and an analysis. After which I will discuss the whole project and conclude this subject.

Literature review

1.1 How soluble content is relevant in research for fertilizers
The longer a piece of land is being exploited as agricultural soil, the lesser nutrients can be found left in the ground. Nutrients can be defined as substances which are needed for life and growth. As time passes and people grow crops, the amount nutrients left in the ground decline. This phenomenon is known as human induced soil degradation. Human induced soil degradation leads to a loss of the global net primary production. This means that the decline of soluble nutrients in agricultural soils reduces the efficiency of the process of growing crops.

Figure 1 shows the amount of human induced soil degradation. If figure 1 is looked at, at first glance a lot of orange and red could be seen. Mostly in area’s such as South Africa the soil degradation is of a higher severity than other parts of the world, such as the Benelux and France. This has also to do with underdeveloped knowledge about how to keep grounds in their optimal state. But figure 1 just goes to show how too little soluble nutrients in the agricultural grounds influences the function of those area’s.

Organic fertilizers could be a solution for the decline of soluble nutrients in agricultural soils. Soluble nutrients can be defined as nutrients which can be transported through the body or plant by water. Which means all soluble nutrients have to be polar, otherwise they can not be dissolved in water. Plants need different nutrients to grow and just work efficiently. So in order to produce an efficient fertilizer, the chemical composition of the ground has to be researched. When the chemical composition of the land is known and the plant which will grow on it is known as well, the needed additions can be calculated.

This also answers the first subquestion: ‘Why is the soluble content relevant in research for fertilizers?’. The importance of the soluble contents of hay or fertilizers or anything else what could be added to the ground, cannot be underestimated. Because too much or too little of a certain nutrient can be disastrous to the general performance of the ground and the plant, which could even lead to infertility of the ground.

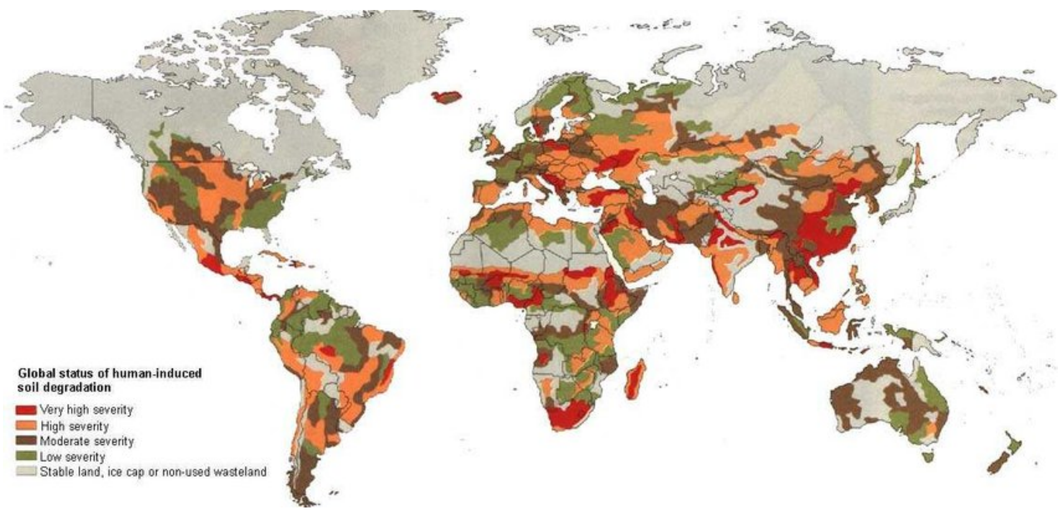


Figure 1 Global status of human induced soil degradation

1.2.1 Soil degradation and its differences

Soil degradation can be defined as the decline of the quality of the soil. Improper use or inadequate management is the reason of this decline. The soils mostly degrading are mostly used for agricultural, urban or industrial use. Soil degradation is highly harmful to the environment and also for human life because soils are fundamental for the production of food and habitation of every organism. Not only is soil degradation harmful for the quality of the soil, the quality of water also declines because of the movement of nutrients and soil with the water. So preventing soil degradation is of big importance for everyone.

Figure 2 Example of soil which has been eroded by water

Examples of soil degradation are: water erosion (see figure 2), wind erosion, salinity, loss of organic matter, fertility decline, soil acidity or alkalinity, structure decline, mass movement and soil contamination. So not every form of soil degradation is happening because of the way humans act. Even though the property- and landowners can not do anything about the way their land change in some cases, it does influence the worth of their property and also how efficient the land can be used. Not only that, but it also unlocks greenhouse gases which are stored in the grounds. This only helps the process of degrading soils which is not a good principle.

1.2.2 How soil degradation influences the economy

The fact that soil degradation influences the amount of crops that can be harvested per acre, also means that the current amount of agricultural grounds can not be used as efficiently as before. Furthermore does the net primary production also decrease when the amount of soil degradation increases. The effect of a decreasing efficiency of the agricultural grounds does not only affect the amount of produce the farmer is able to sell, and with that the farmer’s salary, but also secondary effects of soil degradation which influences society.

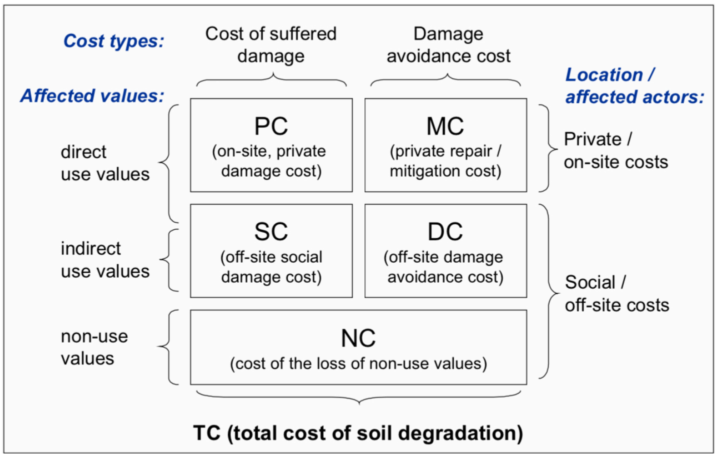


Figure 3 Costs of soil degradation

On site costs of soil degradation are defined by the amount of loss led by the farmer himself. These losses are mostly made up of the loss of value of the land, because the land is less fertile as before which means it is worth less. The MC (private repair/mitigation cost) is made up mostly of costs to limit the damage from the degrading soil, because bringing the soils back in their former pristine condition is in most cases impossible. Most of the time the private costs of soil degradation is fairly manageable. But also those costs are different per country and per region. In some countries the loss led by the farmer are somewhat manageable, with losses between 0-1%. In other regions or countries, because of regional differences in soils, weather and other external factors, the losses led are sometimes more than 5%. With those numbers, prevention of soil degradation is very necessary.

Another research has led to the discovery of the costs globally of water erosion. The lost soils influence the food production of the world. 33.7 million tonnes are lost through the eroding soils which leads to an increase of food prices of 3,5%. This means the global bill is around $8.000.000.000,- per year. As the demand for food grows the losses led by soil degradation will only increase if nothing will be done against it.

1.2.3 Prevention of soil degradation

Soil degradation, as mentioned before is not something that is ideal for the farmer and/or the owner of the land. So protective measures are a welcome addition to the way land is managed. There are lots of ways how to prevent the soil from degrading. Erosion through wind and water are mostly the sorts of erosion which will be prevented.

The basic principle of protecting land is to put a layer of something on top of it. The most natural way to do this is to plant a cover crop. 'Annual grasses, small grains, legumes and other types of vegetation planted to provide a temporary vegetative cover. Cover crops are often tilled under serving as a "green manure" crop.' - University of Rhode Island. Also other natural covers for the grounds can be used to reduce soil erosion. But not only covering the land with (natural) cover can be done to reduce soil erosion, also improving drainage is a way to reduce water erosion.

Soil degradation in the form of decreasing amount of nutrients in the ground is harder to counter. As the contents of the soil decrease, the efficiency of the soil decreases as well. (Organic) fertilizers can certainly help to let the crops grow better, but to get the soil back in its pristine state is nearly impossible.

1.3 What function certain soluble nutrients have

When the relation between particle size and soluble nutrients is analyzed, the need for knowing what function all soluble nutrients have in the ground is something which should not be forgotten about. Upon closer examination the soluble contents found in hay are mostly ions which have several function in the plant and also influence the growth of the plant when added to fertilizers.

As plants have several different processes going on in them to survive, grow and produce fruits etc. Different cations and anions are needed for their respective process. As growth of the plant is a very important aspect several different ions are needed for that process. Chloride, nitrate, nitrite, phosphate, sulphate and ammonium are ions that are vital to the growth of the plant. The regulation of water through the plant is also needed for survival, otherwise parts will dry out and die off. This process will be referred as as osmosis. Potassium and sodium play an important role in osmosis.

The most common ions used in fertilizers are phosphate and nitrogen, as they play a very important role. The reason why nitrogen plays such an important role in the plant is because it is vital in the process of photosynthesis and is a major component in amino acids. When the photosynthesis process can not take place, the plant will eventually die. Nitrogen (N2) as an element itself is useless to plants and has to be bound with oxygen to be absorbed by the plant and be of use. Molecules with nitrogen in them are also the nutrients of which fertilizers consist mostly of.

Several nutrients don't have a single use in the plant. Growth and osmosis are two of the most important processes, but a lot of other ions have other (less) important uses in the plant. Calcium for example is needed for building cell wands and cell membranes and is therefore needed in a large quantity. Another example of a (less) important ion is magnesium. Several enzymes in the plant cannot function without magnesium as cofactor.

A part of the process of synthesizing ATP (Adenosine Triphosphate) is the Krebs-cycle. In the Krebs-cycle a lot of organic acids are part of the process. These organic acids are just like ions able to dissolve in water. But when these acids are in the ground, living plants have no use for them as they can not be absorbed. The molecules are too big and the plants have to make those acid themselves if they want to use them.

When there is shortage of a certain nutrient, a certain process can not take place as it should or just don't take place. Too much of certain nutrients can also be fatal to plants. So keeping a certain amount of nutrients in the ground is of great importance for the growth and survival of plants and crops. This also answers the second subquestion: 'How do cations and anions influence the growth of plants and the efficiency of agricultural soils?'

Materials and method

For this experiment I went to Wetsus European centre of excellence for sustainable water technology. All materials and technology I used for this experiment are in the Wetsus laboratories.

Experiment 1: 'The influence of particle size on soluble content'
Materials for the samples:
- Welkoop 'Pet's own choice' Premium Hooi 2kg
- Milli-q purified water
- Retsch GM 300 grinder
- Fritsch analysette 3 spartan pulverisette 0 sieve, with sieves varying from 0.053 to 7,1+ mm.
- Sartorius balancer
- IKA Loopster digital rotator
- Big centrifuge
- 50 ml sample tubes
- 15 ml sample tubes
- Cation, IC Anion and LC-MS/LC-OCD vials + corresponding caps
- Syringes
- Pipet and pipet tips
- 0.45 µm filters

Analyzers for results:
- Metrohm 761 Compact Ion Chromatography (IC)
- HPLC analyzer

Method: _Note: for the most precise results, more sets of samples have to be made and analyzed._
- Grind the hay in the grinder. With the grinder set as follows:
  Time: 0.20 m:ss
  Speed: 4000 rpm
  Rotating direction: —>
  Interval: 0.05 m:ss
  Direction reversal: Off

  Note: when the grinder is used with different settings, the division of particle sizes changes too.
- Stack the sieves. From top to bottom: 7,1+ mm / 4-7,1 mm / 4-2 mm / 2-0,4 mm / 0,4-0,25 mm / 0,25-0,053 mm. Put the stacked sieves on the tray of the automatic sieve. Secure the stacked sieves and put the settings as follows:
  Time: 10 minutes
  Amplitude: Not too high but the particles will have to fall down
- Put the sieved hay in different packagings, and mark them with the corresponding sizes.
- Put as close as 1 gram of hay in a 50 ml sample tube using the balancer. Repeat this step for every particle size in tubes of their own. Mark them with the corresponding sizes.
- Pipet 20 ml of Milli-q water in every sample tube. Do this by using the pipet two times each tube, with exactly 10 ml of water every time.
- Place all samples in the digital rotator to mix thoroughly and put the settings as follows:
  Time: 30 minutes
  Speed: 70 rpm

- Take the samples out of the rotator.
- Put the samples in the centrifuge (make sure the centrifuge is balanced!). And let the samples centrifuge for at least 15 minutes. When the samples are taken out of the centrifuge, the hay should be at the bottom of the sample tubes. If not, put them back and let them centrifuge for a longer period of time.
- Put the solution in a new 15 ml sample tube. Repeat for every sample. For this step use the pipet, with a new pipet tip for every sample.
- Dilute every sample. For every part sample add 9 parts Milli-q water. *Note: for the results I also made analyzable samples from the samples of the biggest three particle sizes, I did not dilute those.*
- Take a syringe and take some of the diluted sample out of the sample tube. Put a filter on the top of the syringe and fill all three sorts of vials with every sample. In total 18 samples should be made. Put the corresponding caps on the vials and put them in the refrigerator.
- Put the samples in the right analyzers and the results will come on the computer.

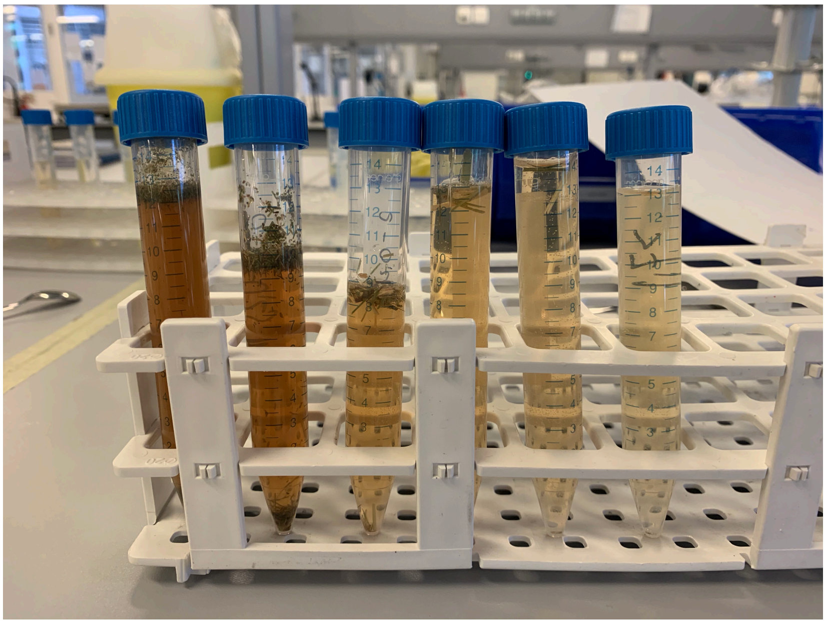


Figure 4: Colour change in the solutions small particles to big particles from left to right

Experiment 2: 'The influence of particle size in soil'
Materials for the samples:

- Welkoop 'Pet's own choice' Premium Hooi 2kg
- Pokon Bemeste Tuinkompost 40 liters
- Soil, can be (almost) any kind
- Plastic pots, at least 2 liters capacity each
- Sieves varying from 7,1 to 0,25 mm
- Retsch AS 200 sieve
- Oven to dry compost
- Petri scales
- Retsch GM 300 grinder
- Sartorius balancer

Method: *Note: for more precise results, more samples should be made and analyzed.*

- Take a sample of the compost and dry the whole sample in the oven.
- Stack the sieves on the Retch plate. The smaller sieves should be placed on the bottom. Put the dried compost in the top and sieve with the following settings:
  Amplitude: 50
  Time: 5 minutes
- Separate every particle size in different petri scales and weigh every sample. This defines the ratio of every particle size in the compost, and also in what ratio's the hay should be ground and put together.
- Put the hay in the grinder and grind as much hay as needed to get enough of every particle size after sieving to make a mix of all sizes to match the ratio's of the compost.
- Dry and sieve the soil, enough to put at least two liters in 9 pots.
- Put the dry masses in the pots as follows:
  1,2,3: only soil
  4,5,6: soil and hay (try to evenly spread the particle sizes)
  7,8,9: soil and dried compost

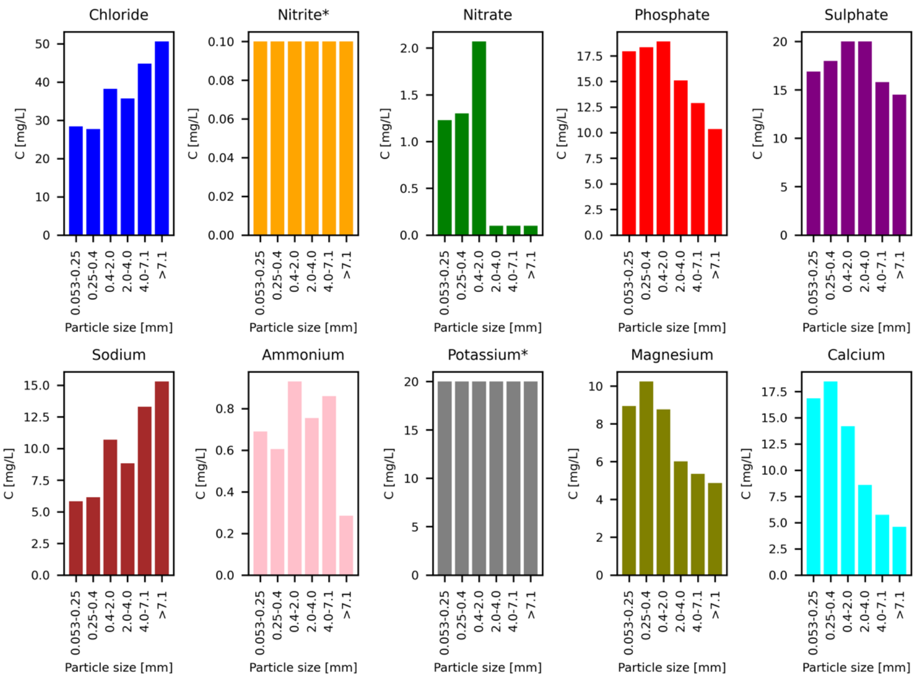
Note: make sure the volumes of the total mixtures are equal and the masses of the hay and dried compost are equal. In my experiment I put a total of two liters of dry substance in the pots.

- Water each pot 500 ml of tap water on the first day, and 300 ml of tap water on the second and third day. Make sure the top layer of the soils are moist for the rest of the experiment.
- After a determined amount of time measure how much time every sample takes to absorb 100 ml of tap water and how much water every pot can hold. For this experiment I took a month before getting the results.

Results and conclusion

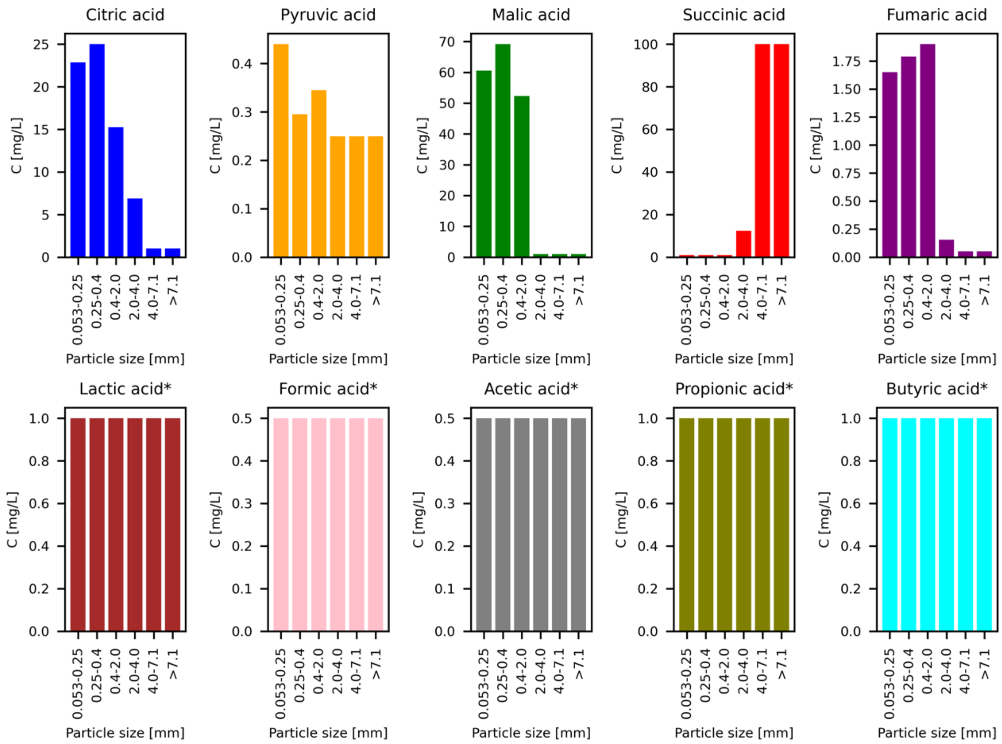
Experiment 1:
Samples
The size of the hay particles varies from 0.053 mm to +7.1 mm. The hay is ground and sieved when dry. All the samples contain 1 gram hay in 20 ml water. The samples are diluted with 10 ml water (table 1 and 2). If the samples are not diluted the concentrations are above the detection limit of the technique. The samples in the tables 3 and 4 are not diluted. Most of the concentrations in these tables are above the detection limit. The concentrations too high for the detection of the technique are indicated with >. The concentrations too low for the detection of the technique are indicated with <. In the graphs, the results too high or too low for the detection limits are marked with *. The size of the hay particles in the not diluted samples are 2.0-4.0 mm, 4.0-7.1 mm and +7.1 mm. The diluted samples are shown in the tables 1 and 2. Some of the concentrations are still too high or too low. The detection limits of the techniques are shown in the tables 5 and 6.

Ions
When looked at graph 1 a few notable elements can be seen. Chloride and sodium have a relatively equal trend, lower concentration with smaller particle size. This is against the hypothesis and most of the other graphs. Calcium, magnesium, phosphate and nitrate have a mostly downward trend which means a higher concentration with smaller particle size. This matches with the hypothesis and the colors of the samples when looked at the colour of the solution after centrifuging. Ammonium and sulphate peak at the middle sized particles, with lower concentrations at the bigger particle sizes so either the results are not as exact as they should be or another factor plays a part in this. Nitrite and potassium have results outside of the detection limits so nothing valuable can be said about those.



Graph 1: soluble concentrations of ions out of hay

Acids
Citric, pyruvic, malic and fumaric acid show a clear downward trend in graph 2. A higher concentration forms through smaller particle sizes which matches exactly with the hypothesis. Succinic acid shows the complete opposite, just like chloride and sodium. Lactic, formic, acetic, propionic and butyric acid all have results outside of the detection limit. So most of the trends are downwards which means the hay has higher soluble content, the smaller the particle size. This also matches with the observations done with the samples.



Graph 2: soluble concentrations of acids out of hay

Conclusion

The research question was: 'In what way does soluble fraction of nutrients change after size reduction?' I expected that the smaller particles will have a higher soluble content. This hypothesis is for the most concentrations correct. The first differences could already be seen when adding the water to the hay. The water with the smaller particles became browner than the water with the larger particles. When you take a look at the graphs, you will see most of the trends go down. This means that the concentration of ions and acids is lower with larger particle size. There are some exceptions. At a few concentrations, no difference can be seen or the concentration even increases. The reason that the concentration of ions and acids is higher with the smaller particles is that the smaller particles have a larger total surface area. As a result, the smaller particles dissolve more ions and acids in the water.

Experiment 2:

Observations

When the pots are watered for the first time, the absorption-time had relatively big differences between the samples. The pots with the hay-mixture absorbed the same amount of water much quicker than the other pots with the more dense mixtures (on top). So in dryer periods of time when the top layer is completely dried out the soils with bigger particles on top can absorb the water far more effectively. Also, after a certain amount of time a fungal culture originated on top of the sample with the hay mixture. Also, the pots with soil only and compost mixture take a lot more time to absorb the entire added amount of water in the first two days than the hay mixture pots.

Setup

Characteristics of the dried compost after sieving

Particle size	Mass
> 7,1 mm	8,794 g
7,1 - 4,0 mm	9,589 g
4,0 - 2,0 mm	14,098 g
2,0 - 0,4 mm	38,522 g
0,4 - 0,25 mm	7,053 g
< 0,25 mm	4,081 g

Characteristics of the hay mixture (the masses of the dried compost times 3)

Particle size	Mass
> 7,1 mm	26,50 g
7,1 - 4,0 mm	28,73 g
4,0 - 2,0 mm	42,31 g
2,0 - 0,4 mm	115,57 g
0,4 - 0,25 mm	21,36 g
< 0,25 mm	12,17 g

Soils and mixtures in pots

1,2,3: only soil
4,5,6: soil and hay mixture
7,8,9: soil and dried compost
Note: the volumes of all mixtures are a total of two liters and the masses of the compost and hay are equal.

The pots are only watered the first three days in these amounts:

- Day 1: 500 ml
- Day 2: 300 ml
- Day 3: 300 ml

The water used is regular, cold tap water.

Results:

Pot number and contents	Absorption time 100 ml water (Min, sec, millisec)	Maximum absorption (after adding 300 ml of water)
1 soil	04:18,20	169 ml
2 soil	05:24,32	177 ml
3 soil	04:32,91	188 ml
4 soil + hay (big top particles)	01:19,38	208 ml
5 soil + hay (smaller top particles)	01:08,58	146 ml
6 soil + hay (small top particles)	09:02,66	112 ml
7 soil + compost	05:59,13	158 ml
8 soil + compost	07:09,46	110 ml
9 soil + compost (big cracks in surface area)	01:49,56	128 ml

Conclusion

Although the results vary a lot in the measurements, together with the observations an accurate conclusion can be formed. The average absorption time of the soil and hay mixture is lower than the other mixtures when looked at the measurements. This also matches with the observations done when watering the first times. Especially when looked at the pots with the bigger particle sizes on top, a significant decrease in absorption time can be seen. This has to do with the bigger surface area the water can be absorbed in, hence the low absorption time of pot number 9 because it had big cracks at the surface area. When looked at the maximum absorption the more dense mixtures hold more water than the mixtures with bigger particles. This also matches the observations of less water coming out of the bottom of the pots when watered than the pots with both mixtures. The absorption times however, are much higher than those with mixtures so when a piece of land is very dense at the top layer the water coming on will not be absorbed as quickly but the land will hold the water much better.

Compost and hay also differ a lot from each other. Both materials add a lot of soluble nutrients to the soil but still differ. One of the biggest advantages of hay is the big decrease in absorption time. When looking at optimization of the soils hay and compost serve the same purpose but also the situation and circumstances. Another big difference between compost and hay or other dried organic material is the need of water. When organic material is added to the soil without the transformation to compost, less water is needed to get soluble content in the soil than when water is used to turn organic matter into compost. After a certain amount of time the dried organic material will also rot and turn into compost when added to the soil.

The big differences in absorption times can be explained by the vast amount of variables in this experiment. Although the circumstances were equal for all pots, the amount of cracks and formation of fungi were vastly different in each pot. Also the lack of plants distorts the results a bit because the research is meant for agricultural grounds which will not be empty when these techniques can be implemented. In short, the bigger the particle size at the surface layer the faster water will absorb. Furthermore, the denser the soil the better the soil retains water.

Applications

The conclusion of both experiments are fairly clear. The smaller the particle size, the higher the concentration soluble content and the smaller the particle size the slower soil absorbs water but the better it retains water. This principle can be applied in a vast amount of situations.

Looking back on the problem of growing populations and degrading soils this information can be really helpful. Depending on the situation and the perceived problem, optimization of the grounds with dead organic material can be possible. For example in a dry area with little precipitation it is important to use as much of the water as possible with highest efficiency. A solution can be to add for example hay or other dead organic material to the top layer of the soil to create the benefit of faster absorption. Not only will the soil be absorbing water more easily, (an)organic content will also be added to the soil which will only improve the growth of the plans even further. In this way the total yield of an acre of soil will be improved which also helps solving the problem of degrading soils and the growing need of food.

Of course the plants growing on the grounds would also influence the characteristics of the land and how the land reacts to changes in the environment. But with more soluble nutrients in the soil, plants grow better which also improves the growth of roots of the plants which improves the structure of the land. This also improves the water retention of the land which leads to a land that needs less water to get equal yield.

The usage of dead organic material as a fertilizer or for optimization of the characteristics of soil is a little obsolete comparing to factory produced artificial fertilizers. The concentrations of the soluble content coming out of those fertilizers are too high to compete. But in countries or area's where resources or technique are limited the usage of dead organic material could make a difference. Not only for farmers but also for the (local) economy, as mentioned before. Also adding the dried organic material to the water improves the nutrients in the soil after adding the water to the land. This also improves the growth of the plants and with that the yield of the acre of land.

So depending on the situation dried organic material can be really helpful in optimizing soils and getting maximum yields. Of course every situation and problem is different so has to be looked at separately but when using the dry organic material in its different fraction sizes an (significant) improvement can be achieved. That's why size matters.

Discussion

The results of both experiments came out pretty much as expected. Even though the results were as expected a vast amount of elements have enough room for improvement. Both experiments should have been done in a greater amount of times. In this way the big differences between the results would be smaller and a better picture could be formed of how everything works and more accurate conclusions could be formed. Also the experiments could be performed more precise, which would also give more accurate results.

Some of the results show the exact opposite of the principles proven in the experiments. I suspect the vast amount of variables have caused these differences between the results. When both experiments would be performed more times and more precise I suspect all results to match the principles and the hypothesis. Two or three measurements have proven to not provide enough information to give a realistic picture of the principles.

For further research I would suggest looking at the optimum amount of soluble nutrients in soils depending on the crop that would grow. After which the principles should be tested in practice. Based on these experiments and the outcome of further research the researched techniques could be used in area's with limited resources to still get enough yield from the agricultural lands and to at least slow down the degradation of the soils.

Of course the COVID-19 virus limited the amount of work that could be done for these experiments in the laboratories. Although it was though sometimes to get the accurate results, the project gave satisfactory results and gave a good picture of how the principle works.

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Tables

Table 1

1g in 20 ml (water as extractant)

1 ml of 10 ml (dilution)

	Cloride mg/L	Nitrite mg/L	Nitrate mg/L	Phosphate mg/L	Sulphate mg /L	Sodium mg/L	Ammonium mg/L	Potassium mg/L	Magnesium mg/L	Calcium mg/L
0.053-0.25	23 33.8	<0.10 <0.10	0.94 1.52	14.9 >20.0	13.8 >20.0	4.84 6.84	0.55 0.83	>20.0 >20.0	7.08 10.8	13.7 >20.0
0.25-0.4	22.9 32.4	<0.10 <0.10	0.84 1.75	15.7 >20.0	16 >20.0	5,05 7.24	0.47 0.74	>20.0 >20.0	8.18 12.3	16.9 >20.0
0.4-2.0	38 38,3	<0.10 <0.10	1.98 2.16	19 18.8	>20.0 >20.0	10.6 10.8	0.94 0.92	>20.0 >20.0	8.72 8.82	14.1 14.3
2.0-4.0	35.7 35.7	<0.10 <0.10	<0.10 <0.10	15.1 15.1	>20.0 >20.0	8.78 8.87	0.8 0.71	>20.0 >20.0	6.04 5.98	8.6 8.6
4.0-7.1	44.8 44.8	<0.10 <0.10	<0.10 <0.10	12.9 12.9	15.8 15.8	13.3 13.3	0.85 0.87	>20.0 >20.0	5.33 5.39	5.71 5.77
+7.1	50.6 50.6	<0.10 <0.10	<0.10 <0.10	10.3 10.4	14.5 14.5	15.3 15.3	0.27 0.3	>20.0 >20.0	4.88 4.86	4.7 4.5

Table 2
1g in 20 ml (water as extractant)
1 ml of 10 ml (dilution)

	Citric acid	Pyruvic acid	Malic acid	Succinic acid	Fumaric acid	Lactic acid	Formic acid	Acetic acid	Propionic acid	Butyric acid
0.053-0.25	15.6 30.1	0.63 <0.25	52.1 69	<1.00 <1.00	1.3 >2.00	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00
0.25-0.4	19.4 30.6	0.34 <0.25	58,8 79.5	<1.00 <1.00	1.58 >2.00	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00
0.4-2.0	15.2 15.3	0.44 <0.25	55.7 49	<1.00 <1.00	1.87 1.93	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00
2.0-4.0	6.31 7.44	<0.25 <0.25	<1.00 <1.00	13.4 11.3	0.16 0.15	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00
4.0-7.1	<1.00 <1.00	<0.25 <0.25	<1.00 <1.00	>100 >100	<0.05 <0.05	<1.00 <1.00	<0.50 <0.50	<0.51 <0.52	<1.00 <1.00	<1.00 <1.00
+7.1	<1.00 <1.00	<0.25 <0.25	<1.00 <1.00	>100 >100	0.05 0.05	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00

Table 3
1g in 20 ml (water as extractant)

	Cloride mg/L	Nitrite mg/L	Nitrate mg/L	Phosphate mg/L	Sulphate mg /L	Sodium mg/L	Ammonium mg/L	Potassium mg/L	Magnesium mg/L	Calcium mg/L
2.0-4.0	>80.0 >80.0	<0.10 <0.10	>20.0 >20.0	>20.0 >20.0	>20.0 >20.0	>40.0 >40.0	8.05 8.15	>20.0 >20.0	>20.0 >20.0	<0.10 <0.10
4.0-7.1	>80.0 >80.0	<0.10 <0.10	>20.0 >20.0	>20.0 >20.0	>20.0 >20.0	>40.0 >40.0	6.92 7.05	>20.0 >20.0	>20.0 >20.0	>20.0 >20.0
+7.1	>80.0 >80.0	<0.10 <0.10	>20.0 >20.0	>20.0 >20.0	>20.0 >20.0	>40.0 >40.0	6.06 6.1	>20.0 >20.0	>20.0 >20.0	>20.0 >20.0

Table 4
1g in 20 ml (water as extractant)

	Citric acid	Pyruvic acid	Malic acid	Succinic acid	Fumaric acid	Lactic acid	Formic acid	Acetic acid	Propionic acid	Butyric acid
2.0-4.0	>100 >100	<0.25 <0.25	>100 >100	>100 <1.00	>2.00 >2.00	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00
4.0-7.1	100 97.4	<0.25 <0.25	>100 48.8	<1.00 >100	>2.00 >2.00	<1.00 <1.00	<0.50 <0.50	<0.51 <0.52	<1.00 <1.00	<1.00 <1.00
+7.1	>100 95.3	<0.25 <0.25	>100 94.1	<1.00 >100	>2.00 >2.00	<1.00 <1.00	<0.50 <0.50	<0.50 <0.50	<1.00 <1.00	<1.00 <1.00

Table 5
metrohm 761 compact ion chromatography (IC)

	Lower mg/l	Upper mg/l
Chloride	0,1	80
Nitrite	0,05	20
Nitrate	0,1	20
Phosphate	0,05	20
Sulphate	0,05	20
Sodium	0,1	40
Ammonium	0,1	20
Potassium	0,1	20
Magnesium	0,1	20
Calcium	0,1	20

Table 6
Shimadzu TOC Analyser

	Lower mg/l	Upper mg/l
Citric acid	1	100
Malic acid	1	100
Succinic acid	1	100
Lactic acid	1	100
Propionic acid	1	100
Butyric acid	1	100
Formic acid	0,5	100
Acetic acid	0,5	100
Pyruvic acid	0,25	20
Fumaric acid	0,05	2