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Natural Flocculant from a Combination of *Moringa oleifera* Seeds and Cactus Cladodes (*Opuntia ficus-indica*) to Optimize Flocculation Properties

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Abstract: The lack of access to clean water worldwide and organic, inorganic as well as biological contamination of existing freshwater sources are a major problem for around 2 billion people, especially in the countries of the global south. One sign of polluted water is turbidity. It is generally caused by colloidal and particulate suspended solids. Chemical flocculants are often used to reduce turbidity and thus eliminate the mostly harmful substances that cause it. However, these have some disadvantages, such as cost and availability, so increasingly natural plant-based flocculants are coming into focus and are considered as an alternative option. In this study, Moringa seeds (*Moringa oleifera*) and cactus cladodes (*Opuntia ficus-indica*) were investigated as innovative and environmentally friendly flocculants for water treatment. The parameters investigated included absolute turbidity reduction and flocculation activity, as well as shear strength of the resulting flocs. The flocculation experiments were conducted as simultaneous tests in beakers. Experiments were conducted using both a laboratory-prepared model suspension with an initial turbidity of approximately 139 NTU and natural surface water with an initial turbidity of approximately 136 NTU. The flocculant dosages used ranged from 100 to 300 mg/L. The results show that although Moringa seeds had the highest flocculation activity (up to 93%), the flocs were very fragile and were destroyed again even at low induced shear forces. Flocculants from cactus yielded stable flocs, but the flocculation activity (maximum at 54%) was not as high as that of Moringa. The combination of the two materials resulted in a flocculant with sufficiently high flocculation activity (76%) and stable flocs, which could withstand higher shear forces potentially induced in further treatment steps.

Keywords: cactus; *Moringa oleifera*; natural flocculant; shear strength; turbidity reduction; water treatment

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1. Introduction

Around 2 billion people worldwide do not have access to clean water. Of these, about 785 million people do not have even a basic drinking water supply [1]. However, access to clean and safe water is essential for survival and development. It is also a human right and enshrined as Goal 6 "Ensure access to water and sanitation for all" in the United Nations (UN) Sustainable Development Goals (SDGs) [2]. Clean water is not only important for health, but also essential for reducing poverty, ensuring peace, and respecting human rights. Many countries face challenges such as strong population growth and thus increasing demand for clean water, water scarcity due to overexploitation, water pollution, and advancing climate change [3]. Approximately 114 million people rely on drinking untreated surface water [4].

Flocculation is a treatment method for removing suspended and colloidally dissolved pollutants from water. It is used to coagulate suspended particles, including microbes, to improve their sedimentation [5]. Chemical flocculants are usually used to purify turbid surface water. However, these are rarely available in rural areas and are often not affordable for local people [6]. In addition, chemical flocculants can potentially have negative impacts on people's health and the environment. They also generate a large amount of sludge that is costly to treat and dispose of [7]. Natural flocculants are of great interest in this regard because they are natural and inexpensive products that are environmentally friendly and considered safe for human health [8]. Natural flocculants are also suitable for household use. According to [5], household water treatment approaches have the potential to have rapid and significant positive health effects, especially in situations where a piped water system is not available and people rely on other water sources that may be contaminated. Household water treatment can also serve to address the widespread problem of microbially unsafe piped water supplies [5]. The most studied natural flocculant to date is the seed of the Moringa tree [9]. In various studies [9–15], Moringa seeds were found to remove turbidity by 90% to 99% and thus can be a possible substitute for conventional flocculants. However, none of the existing papers dealing with the use of *Moringa oleifera* as a flocculant in drinking water treatment address the shear strength of the resulting flocs. Further literature searches also failed to find hardly any relevant publications dealing with the shear strength of natural flocculants, especially *Moringa oleifera*.

Flocculation tests carried out with Moringa seeds because of this have shown that the flocs formed are very fragile, irrespective of the flocculation conditions and the water samples used, and disintegrate again even at low induced shear forces. This is particularly problematic for further processing steps such as filtration, sedimentation or flotation, as shear forces always act on the flocs due to the turbulence in the water. Flocculants from Moringa are therefore only suitable for water treatment without restrictions in individual cases.

In the course of further research work, other natural flocculants were also investigated and a high shear strength of the resulting flocs was observed in the case of flocculants from cactus; however, the flocculation activity was not satisfactory as residual turbidity remained too high in the supernatant. Based on these observations, two series of experiments were carried out to investigate whether a combined flocculant from Moringa and cactus could exhibit good flocculation activity and produce flocs that can withstand high shear forces. The first series of tests was carried out with artificially produced surface water in order to be able to exclude effects of ingredients of natural surface waters on the flocculation process. In the second series of tests, samples from a natural surface water were used to confirm the real use of the new flocculant on a laboratory scale.

2. Materials and Methods

2.1. Surface Waters Used

In the first series of experiments, the removal of turbidity by natural flocculants was investigated using an artificially prepared surface water. For this purpose, ten liters of the model suspension were prepared before the start of the experiment, the ingredients and concentration of which are shown in Table 1. The suspension was then stirred on a magnetic stirring plate for at least 30 min to ensure that the solids dissolved as far as possible and the substances, such as the bolus contained, were sufficiently hydrated.

In the second series of experiments, samples were taken from a local stream called Mühlbach and used for the flocculation experiments in order to test the flocculants under conditions that were as real as possible. The water analyses can be viewed on the HLNUG data portal at the following link: https://www.hlnug.de/messwerte/datenportal/messstelle/4/6/180/ (accessed on 20 May 2022).

Table 1. Composition of the model suspension.

Chemicals Used	Producer	Concentration (in g/L)
Calcium carbonate	Merck KGaA, Darmstadt, Germany	0.1
Potassium sulfate	AppliChem, Darmstadt, Germany	0.025
Bolus	Carl Roth GmbH, Karlsruhe, Germany	0.05
Silica	Carl Roth GmbH, Karlsruhe, Germany	0.0015
Sodium chloride	Honeywell, Seelze, Germany	0.1
Sodium hydrogen carbonate	Carl Roth GmbH, Karlsruhe, Germany	0.1
Clay powder	Terra Exotica, Alfeld, Germany	0.05
Calcium nitrate	Carl Roth GmbH, Karlsruhe, Germany	0.03

2.2. Production of Flocculants

For the production of a flocculant from cactus, some shoots were cut off. The preparation of the powder was similar to that described in [16–18]. First, the fresh cactus leaves were washed with distilled water to remove dirt. Then, the cactus shoots were crushed and dried in a drying oven at 80 °C for about 12 h. During this process, the cactus lost almost 44% of its weight. Subsequently, the cactus was crushed with a mill and finally sieved through a mesh size of 125 µm. Figure 1 shows the process in pictures. The powder was then prepared with a solution of 0.5 mol/L NaCl and stirred for ten minutes.

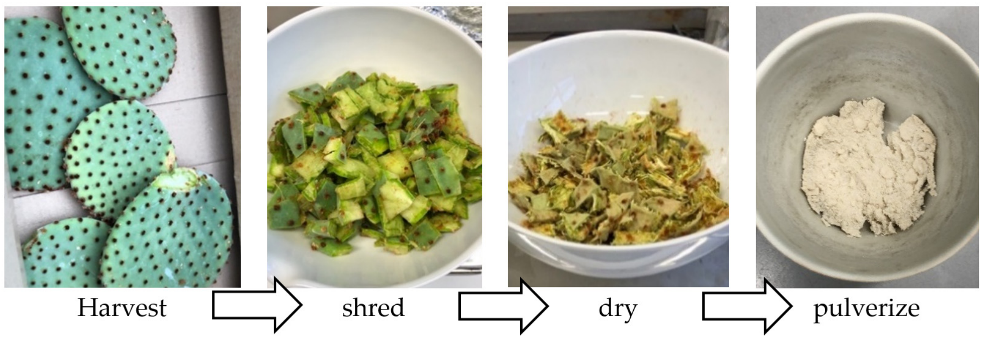


Figure 1. Preparation of a flocculant from cactus.

For the combined flocculant from Moringa seeds and cactus, the dry Moringa seeds were first pulverized. The powder from the Moringa seeds and that from the cactus cladodes were then mixed together in different weight ratios and a 0.5 molar NaCl solution. The flocculant was then stirred on a stir plate for ten minutes and filtered through a filter cloth.

2.3. Jar Test

Flocculation tests were carried out in order to investigate the test objectives mentioned at the beginning. The tests were conducted as jar tests in accordance with DVGW Code of Practice W 218 “Coagulation and flocculation in water treatment—Part 2: Test methods”. The raw water was initially stirred at 100 rpm for about five minutes to homogeneously distribute any settled solids. The flocculant was then added and stirred at 100 rpm for one minute. In this coagulation phase, care must be taken to ensure that the flocculant is distributed quickly and homogeneously in the water, since coagulation and thus the formation of microflocs begins directly with the addition of the flocculant. For the subsequent flocculation phase, during which the microflocs can agglomerate into macroflocs, the stirring speed was reduced to 20 rpm. After 15 min, the agitators were switched off. During the subsequent sedimentation phase, the stirrers were removed from the beaker so as not to hinder the sinking of the flocs and prevent their destruction.

2.4. Turbidity Measurement

Turbidity is used as the most important indicator for checking the effectiveness of the flocculants. A turbidimeter, which measures the scattered light component, was used to measure turbidity. A detailed description of the operation of a turbidimeter can be found in DIN EN ISO 7027-1:2016.

Samples were taken below the water surface using a pipette and mixed again before measurement to avoid sedimentation processes distorting the measurement. The reduction of turbidity was calculated according to the following equation:

$$\text{Turbidity reduction } [\%] = \frac{\text{Turbidity}_{\text{initial}} - \text{Turbidity}_{\text{final}}}{\text{Turbidity}_{\text{initial}}} * 100\% \tag{1}$$

From the measured turbidity values, the flocculation activity could also be calculated using the following formula:

$$\text{Flocculation activity } [\%] = \frac{\text{Turbidity}_{\text{final, control}} - \text{Turbidity}_{\text{final, sample}}}{\text{Turbidity}_{\text{final, control}}} * 100\% \tag{2}$$

The value of the flocculation activity, in contrast to the turbidity reduction, allows conclusions to be drawn about the actual additional turbidity reduction by the flocculant, since it includes the final turbidity in the control beaker. That is, the turbidity reduction that occurs by sedimentation alone is also considered and the reduction by the flocculant is assessed in relation to it. For this reason, this value is considered more meaningful than that of the turbidity reduction alone.

2.5. Shear Strength Test

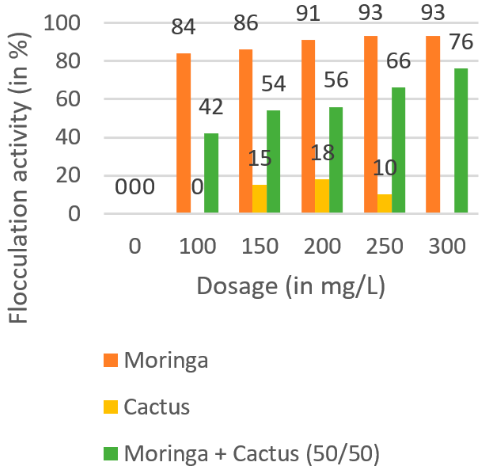
After completion of the previously described test procedure, additional shear tests were carried out. For this purpose, the beaker with the highest flocculation activity was selected for each experiment. In this process, the stirrer was turned on again for one minute and then sedimentation was allowed to resume. The sedimentation time selected was based on the sedimentation time measured in the jar test, during which most of the flocs had settled. After this time, the turbidity in the beaker was measured again. If flocs were mechanically damaged by the renewed energy input, increased residual turbidity resulted. The residual turbidity readings were plotted against the set stirring speeds, giving a visual representation of the shear strength of the flocs. The following stirring speeds were selected for the shear strength tests: 30 rpm, 40 rpm, 50 rpm, 60 rpm, 70 rpm, and 80 rpm.

3. Results

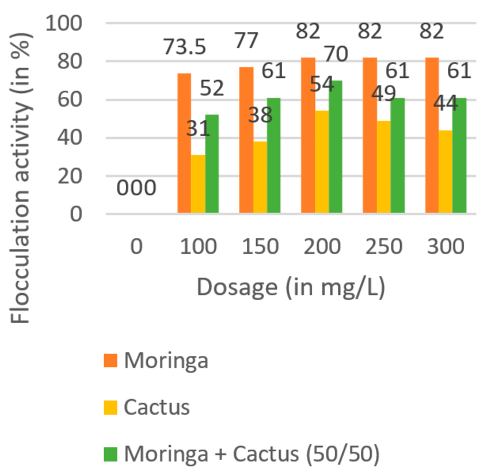
3.1. Flocculation Activity

The following is a comparison between the test results of flocculants from Moringa seeds and cactus cladodes. In this context, the results on Moringa from previous studies conducted by the IWAR Institute of the Department of Civil and Environmental Engineering at Darmstadt Technical University are adopted. In addition, combined flocculants of Moringa and cactus were used in different mixing ratios.

The flocculation activity in the model suspension was a maximum of 93% for the flocculant from Moringa, whereas the flocculation activity in the tests with cactus was a maximum of 18%. The flocculant prepared from both substances again showed a maximum activity of 76%. The flocculation activity data in the model suspension are shown graphically in Figure 2a.



(**a**)



(**b**)

Figure 2. Flocculation activity in model suspension (**a**) and natural surface water (**b**).

Maximum flocculation activity and thus turbidity reduction in natural surface water was achieved using Moringa seeds in combination with 0.3 mol/L NaCl at doses of 200 mg/L, 250 mg/L, and 300 mg/L, as shown in Figure 2b. In these cases, the flocculation activity was 82%; the minimum achievable residual turbidity was 4 NTU. The maximum achievable flocculation activity with cactus at a dose of 200 mg/L was about 54% with a minimum residual turbidity of 18 NTU.

If the flocculants prepared with different mixing ratios of Moringa and cactus are now considered, the flocculation activities shown in Figure 3 are obtained. For this purpose, a flocculant of 100% Moringa and 0% cactus (100/0), a flocculant of 67% Moringa and 33% cactus (67/33), a flocculant of 50% Moringa and 50% cactus (50/50), a flocculant of 33% Moringa and 67% cactus (33/67), and a flocculant of 0% Moringa and 100% cactus (0/100) were prepared and compared in terms of their flocculation activity. All flocculants, except for the one consisting solely of Moringa, were spiked with 0.5 mol/L NaCl. To the flocculant prepared from Moringa, 0.3 mol/L NaCl was added. This had been shown to be the optimum extraction method in previous experiments. The flocculation tests with flocculant from 100% cactus in model suspension showed negligible flocculation activity, which is why they are not shown in the figure. The results in Figure 3 at left show a trend of increasing flocculation activity proportional to the Moringa content of the flocculant used. A similar picture can be seen in Figure 3 at right. The highest flocculation activity in surface water was achieved using flocculant containing the mixing ratio 67/33. Results of experiments with flocculant 67/33 generally showed a relatively uniform, achieved residual turbidity of $\leq$5 NTU at all tested dosages. The results of the flocculant consisting only of Moringa obtained similar results. At dosages between 150 and 300 mg/L, turbidity $\leq$ 5 NTU was also achieved. The determined flocculation activity in model suspension tended to decrease with increasing cactus content in the flocculant. When the cactus content was increased to 67%, only turbidity values between 10 and 15 NTU could be achieved. With a further increase to 100%, the turbidity values were about twice as high (between 20 and 30 NTU).

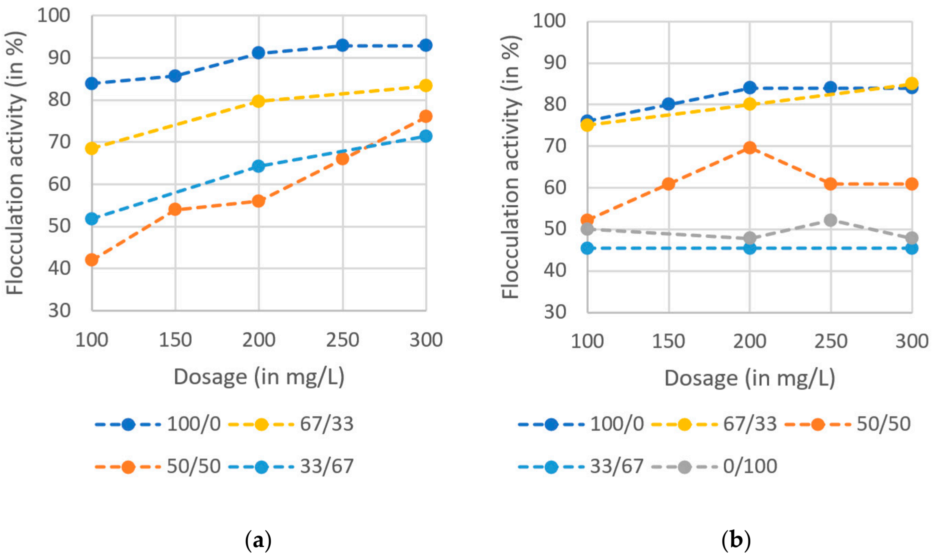


Figure 3. Flocculation activity of flocculants prepared with different mixing ratios of Moringa and cactus in model suspension (**a**) and surface water (**b**).

3.2. Floc Structure and Sedimentation Time

The sedimentation time, during which most of the flocs formed settled to the bottom, changed with different mixing ratios of Moringa and cactus. The flocs sank fastest when flocculant from 100% cactus in surface water was used, which indicated a denser floc structure. As seen in Figure 4, this was also easily confirmed by visual inspection. The resulting flocs of the 100% cactus flocculant were clearly visible at the bottom of the beaker, whereas the flocs of the 100% Moringa flocculant were significantly smaller and, in some cases, even still floating in the supernatant. Already at a concentration of 100 mg/L cactus flocculant, most flocs settled to the bottom within two minutes. Increasing the concentration did not result in any further reduction of the settling time. In contrast, the flocs formed by Moringa required about ten times the time (about 20 to 27 min) regardless of the water used and the applied flocculant dosage. With the mixing ratios 67/33, 50/50, and 33/67, the sedimentation times decreased significantly. Compared to the flocculant made from 100% Moringa, the sedimentation times were at least halved in both test series with model suspension and surface water. Still, there was no clear dependence of the sedimentation time of the formed flocs on the mixing ratio of the used flocculants.

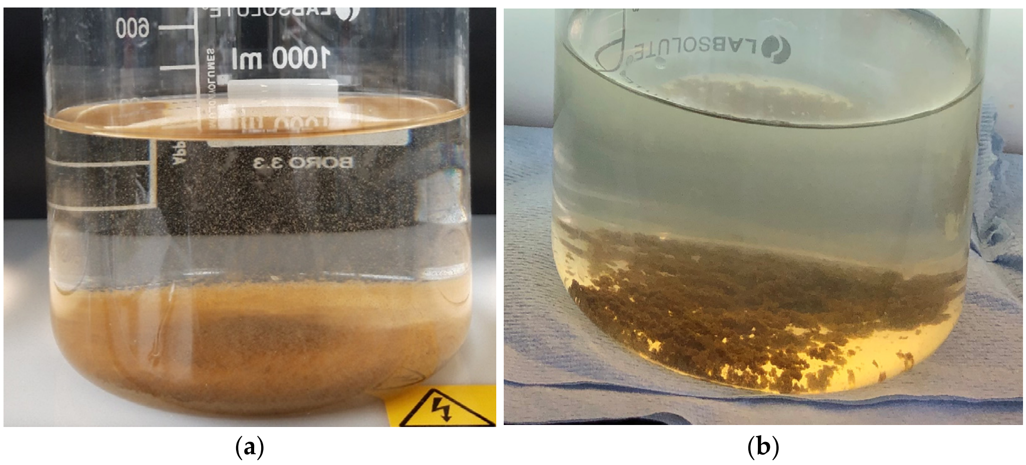
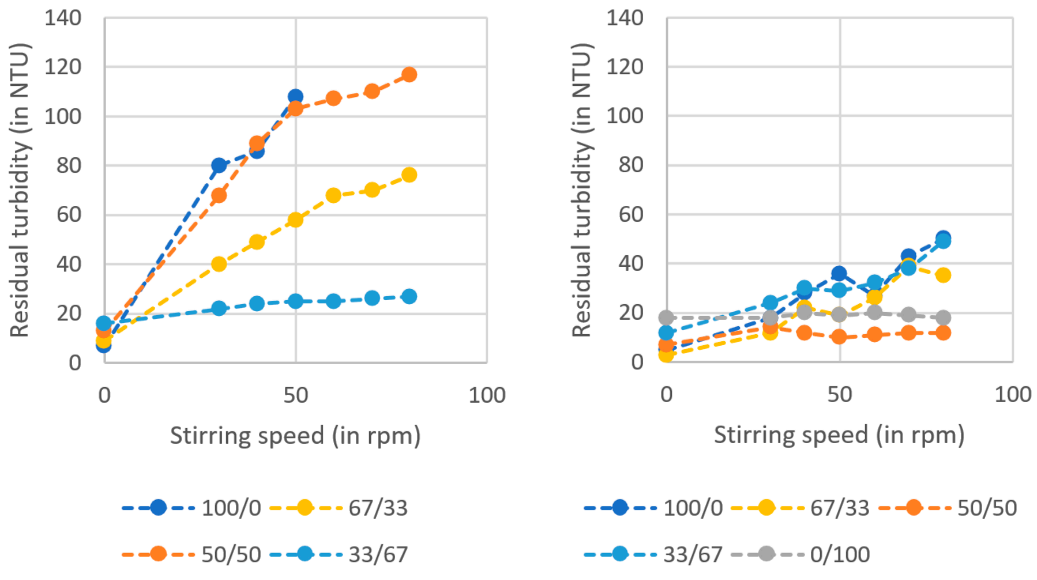


Figure 4. Beakers with flocculating sludge of 100% Moringa flocculant (**a**) and 100% cactus flocculant (**b**) in surface water.

3.3. Shear Strength

Figure 5 shows the residual turbidity measured after the respective energy inputs during the shear strength tests carried out. The results of the shear strength tests in the model suspension are shown on the left. The results of the test series with surface water are shown on the right. In the model suspension, the shear strength test with flocculant from 100% Moringa was already stopped after a stirring speed of 50 rpm, since the turbidity had already reached a value of 108 NTU again; this was close to the initial turbidity and it could be assumed that all flocs had already been destroyed again. Additionally, with flocculant from 100% cactus, no shear strength test could be carried out in the model suspension, as no turbidity reduction in the first place could be achieved.



(**a**) (**b**)

Figure 5. Shear strength of flocculants prepared with different mixing ratios of Moringa and cactus in model suspension (**a**) and surface water (**b**).

Figure 5 on the left shows that the measured residual turbidity with flocculant 33/67 was relatively constant between 16 and 27 NTU. This combination accordingly exhibited the highest shear strength in the model suspension. With the 67/33 combination, the residual turbidity already increased somewhat more steeply. With the 50/50 combination, the turbidity increased from 13 to 68 NTU after only one minute of stirring at 30 rpm, and from 7 to 80 NTU with the 100/0 flocculant.

The shear tests performed in surface water show different results, which can be seen in Figure 5 on the right. In general, the flocs in the model suspension showed a lower shear strength than those in surface water. Here, the 50/50 flocculant showed very little turbidity variation between 7 and 14 NTU. The steepest increase was also observed with the flocculant exclusively from Moringa. Here, the turbidity increased from 5 to 18 NTU after one minute of stirring at 30 rpm. At the end of the shear strength test, the turbidity was about 50 NTU. A similar increase in turbidity was observed with flocculant 33/67. Here, the turbidity increased from 12 to 24 NTU after stirring at 30 rpm. The final residual turbidity after the entire shear strength test was 49 NTU. In natural surface water, the most stable flocs were produced by the flocculant made from 100% cactus, while the residual turbidity was around 20 NTU over the entire shear strength test run.

Although the data from both sets of experiments did not show a clear result, there was a trend that the shear strength of the resulting flocs could be increased by using a combined flocculant of both materials, Moringa and cactus.

3.4. Experimental Conclusions

When comparing Moringa and cactus in terms of turbidity reduction and floc formation, it was found that Moringa could achieve higher turbidity reduction and thus generate lower residual turbidity, but cactus formed much larger and more stable flocs, which formed sediment much faster and were not as fragile. To investigate whether the advantages of the two flocculants could be combined, flocculants were prepared from different mixing ratios of the two materials. In Figure 3, it can be seen that the flocculation activity tended to decrease with increasing cactus content. In the surface water, the 67/33 flocculant even achieved slightly better values than the flocculant prepared from 100% Moringa. It was also notable that the dosages maximizing flocculation activity were no longer 200 mg/L, as was the case independently for cactus and Moringa. The 67/33 flocculant had its optimum at 300 mg/L, the 50/50 flocculant at 400 mg/L, and the 33/67 flocculant was able to achieve the equal turbidity reduction at 100 mg/L, 200 mg/L, and 300 mg/L. These results confirm the first hypothesis that turbidity removal can be improved by combining Moringa with cactus compared to cactus alone.

The second hypothesis concerning the shear strength of the flocs was also conditionally confirmed. As shown in Figure 5, the smallest increase in turbidity of the surface water over the shear strength tests was observed for the 0/100 and 50/50 flocculants. It can be concluded that these were the most stable and shear-resistant flocs. This result can be confirmed by the observed sedimentation time, which was the lowest in these tests. This indicates that the flocs from the two flocculants were particularly dense and therefore both sank quickly and were particularly shear-resistant. As a result, the flocs were easier to separate in subsequent processing steps and less likely to be destroyed. In the shear tests performed, the low shear strength of the flocs of the other combinations was particularly noticeable. Although it was expected that the flocs of 33% Moringa and 67% cactus would have higher shear strength than those of 50% Moringa and 50% cactus, the turbidity at the end of the experiment with flocculant 33/67 was about four times higher than that at the beginning of the shear test. This suggests that existing surface water ingredients hindered ideal floc formation and therefore shear strength was reduced. The shear strength tests in the model suspension unfortunately also did not show a clear linear relationship.

4. Discussion

In contrast to the experiments with surface water, the flocculant of cactus showed very low or no flocculation activity in the model suspension (see Figure 2b). According to the results of [19], one explanation for this is the absence of organic material. The authors concluded that the flocculation process was positively influenced or even enabled by organic material. Flocculation in water with purely mineral sediments would at least result in smaller diameters of the flocs. Since organic material was completely absent in the model suspension, this is most likely the reason why the cactus flocculant did not form flocs and thus showed hardly any flocculation activity in the model suspension. The already low flocculation activity at dosages between 150 and 250 mg/L reduced again to 0% at flocculant dosages of 300 mg/L, which was most likely due to restabilization of the particles. This effect can also be seen in the right part of Figure 2. With cactus, as with combined flocculants, the flocculation activity decreased again from a dosage of 200 mg/L, indicating that the optimum or maximum flocculant quantity had been exceeded and that a further increase would only have detrimental effects. With Moringa flocculant, there was no increase in flocculation activity above 200 mg/L. It can be assumed that at higher dosages than those investigated here, the activity also decreased again for the same reasons described.

The reason why the Moringa flocculant nevertheless exhibited flocculation activity in the model suspension was, in turn, the positive charge of the polyelectrolytes in the flocculant. In contrast to the cactus flocculant, the Moringa flocculant has compounds with cationic groups according to [13–15]. Due to the mostly negative surface charge of the particles contained in the water, the flocculation effect is enhanced by charge neutralization.

The resulting long sedimentation time could be significantly shortened by the addition of cactus. The shortened time indicates more compact, denser flocs with larger diameters, which could also be confirmed by photographic images. Cactus flocculants or flocculants containing cactus produce denser flocs because the flocculation mechanism of bridging is favored by the long-chain polyelectrolytes [16,20]. However, contrary to expectations, no linear dependence of the shear strength of the flocs on the cactus content in the flocculant could be conclusively demonstrated. Due to the use of natural flocculants, it is possible that sometimes more, sometimes less flocculant compound of the materials used was present in the flocculants prepared. Thus, contrary to the expected trend, outliers may occur. This can only be eliminated by further extensive investigations in order to compensate as far as possible for this systematic error by the statistical certainty thus gained.

Based on the findings of the test series shown here in conjunction with the results of [21], it can be concluded that the flocculating active substances of both raw materials used (Moringa and cactus) are cationic polyelectrolytes. Although Moringa forms smaller, weaker flocs, a high flocculation activity is nevertheless achieved. If sufficient organic matter is present in the water sample used, flocculants from cactus also show high activity and form compact, shear-resistant flocs.

In conclusion, no general rule can be derived from this as to which combination of Moringa and cactus is the best, but it can be said that a combination of Moringa and cactus can offer great advantages over Moringa in terms of shear strength and sedimentation time, and over cactus in terms of turbidity reduction. For example, shear strength was improved by about 76% (at 80 rpm) and sedimentation time was reduced by up to 98%. Turbidity reduction was improved by up to 83% compared to cactus (max 54%). Another advantage over the flocculant from Moringa is the size of the flocs that form. Since they are much larger, they not only sink faster, but are also easier to separate. Nevertheless, there is no universal mixing ratio. For each waterway, preliminary tests must be conducted to determine the optimal mixing ratio of *Moringa oleifera* and cactus.

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