



Treatment Protocol Efficiency of Plant Aggregates to Their Influence on Swelling and Shrinkage

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Abstract. The use of plant resources for the development of insulation biocomposites, demands a solid understanding of their morphological, physico-chemical behaviour, and mechanical properties in relation to their conditions of use. The behavior of bio-composites and their durability are influenced by the high sensitivity of plant aggregates to hydro/hydrothermal conditions. This sensitivity of vegetable aggregates, due to water absorption, can be controlled by treatments. Several factors are important to assess the impact of the treatment on the properties of the aggregates. Among them, the used concentration, duration, and temperature. The objective of this research is to adjust the treatment procedure for vegetable aggregates to improve the behavior of the bio-composites. The study is interested in investigating the variations of the treatment duration and the rinsing time affects the aggregates.

Two salts are used in this work, sodium bicarbonate NaHCO_3 and sodium chloride NaCl at a concentration of 10% by weight to evaluate the swelling and shrinking phenomena. After completing the treatment, the aggregates are exposed to wetting and drying cycles to evaluate the treatment's efficiency on swelling and shrinking behavior. During the treatment, pH measurements are taken, and the dimensional variations of the aggregates are analyzed under the microscope.

The pH measurement during treatment shows an increase for NaHCO_3 -treated aggregates compared to NaCl and untreated aggregates. At the same time, the pH measurement during rinsing decreases with the removal of salts after a 20-min rinse for NaHCO_3 versus 5 min for NaCl . Microscopic analysis confirms the effectiveness of NaHCO_3 treated aggregates in decreasing swelling and shrinkage behavior, as well as their durability, as they do not show a decline in effectiveness over time. Finally, it is effective to treat the aggregates with salt for 3 days, dry them at a moderate temperature, and then rinse them for 20 min to remove any remaining salt and impurities.

Keywords: Treatment · vegetable aggregates · swelling · shrinking

1 Introduction

Reducing the environmental impact of construction materials is a key concern in the building and construction industry. In this context, the interest in biobased material is growing for a few years. These materials are considered more sustainable and environmentally friendly than traditional materials [1]. The plant aggregates can guarantee not only the reduction of carbon emissions and energy consumption, but also provide thermal comfort [2]. Thanks to their interesting thermal, hydric and mechanical performances, they qualify as a good insulator for buildings [3].

However, as any building material, they are exposed to various environmental use conditions of long term such as humidity, temperature, or UV radiation. Moreover, studies on plant aggregates have revealed their hygroscopic behavior and high sensitivity to water vapor and contact with liquid water. The ability to absorb moisture help to control the humidity in each environment to maintain the freshness and quality of building comfort. Nevertheless, this affects their behavior by causing damage bio-composites. This consequence is highly related to the dimensional variation which is influenced by hygro and hydrothermal conditions [4]. When plant aggregates are exposed to environmental conditions such as humidity and temperature variations, they are likely to absorb moisture, resulting in swelling [5–7]. When subjected to repetitive immersion-drying cycles, the aggregates experience degradation resulting in a loss of swelling and shrinkage due to the combined effects of moisture absorption and material weakening [8].

To reduce the amount of absorbed water, there are a large number of treatments in the literature depending on the specific problem and the desired result. Treating the aggregates, weather by a chemical or physical treatment improve the mechanical, structural, or hygroscopic properties by modifying their chemical component. In this regard, studies on component modification using salt solutions show their benefit in decreasing their water absorption rate. These modifications are generally based on the use of hydroxyl functional groups capable of changing the composition of the aggregate structure [9]. In this way, the tendency to absorb moisture is reduced. The treatment principle consists in immersing the aggregates in a salt solution, for a studied time, and carry out the rinsing to release the quantity of salt. In their work, Fiore and al. [10, 11] soaks a concentration of 10% by weight of NaHCO_3 for a period of 5 days to treat the sisal fiber in order to improve their tensile and flexural properties. The same concentration was used by dos Santos et al. [12] for a duration of 96 h to treat coconut fibers to increase their mechanical properties. However, for Chaitanya et al. [13], a duration of 72 h presented optimal strength properties for aloe vera fibers. The second salt used to soak the aggregates in the literature is sodium chloride. Some attempts successfully proved the decrease in water absorption by treating the aggregates with NaCl [14] and subsequently their swelling and shrinkage. This is a commonly used salt for clay soils to decrease their liquid and plastic limit and plasticity index [15]. Nevertheless, the above-mentioned authors focused in their studies on the optimization of salt concentration and the effect of exposure time, and none of them studied the effect of rinsing and the duration of treatment and their response on the swelling and shrinking behavior.

For this purpose, an attempt is made to study the efficiency of the treatment protocol on vegetable aggregates. The objective is to propose an experimental protocol for the treatment of plant aggregates with emphasis on the effects of the treatment on their

swelling and shrinkage behavior. This objective is achieved through two processes. The first one consists in studying the influence of the treatment time on the aggregates. The second part of the study is to focus on the influence of the rinsing time to remove salt to reach a stable pH after treatment [16] to avoid their later impact. Therefore, these two processes are monitored by measuring the pH of the salt solutions during the treatment of the aggregates in parallel with the microscopic analysis. The pH measurement allows the identification of interactions between the salt solution and the aggregates. The microscopic analysis confirms the influence of the treatments on the aggregates. Furthermore, the aggregates will be subjected to four cycles of wetting and drying after the end of each treatment to measure swelling and shrinkage to examine the effectiveness of the treatment on the aggregates.

2 Materials and Methods

2.1 Materials

Four types of aggregates are studied in this work, wheat straw, rapeseed straw, pith and sunflower bark and are all collected in the Centre Val de Loire region. These aggregates have been used for biocomposites developed for thermal insulation of buildings in previous work of the team [17, 18]. The aggregates chosen are first cleaned by a dry brushing to remove the dirt and debris from the aggregates. Then, they are cut to obtain a representative sample for microscopic analysis. Through this study, we tested the influence of two salt treatments, sodium chloride NaCl [19] and sodium bicarbonate NaHCO₃, in reducing the rate of water absorption and swelling and shrinkage of aggregates [20]. The salts sodium chloride (NaCl, $M = 58,44 \text{ g/mol}$, $\rho = 2.17 \text{ g/cm}^3$) and sodium hydrogen carbonate (NaHCO₃, $M = 84.01 \text{ g/mol}$, $\rho = 2.21 \text{ g/cm}^3$) are purchased from CARLROTH, a German company.

2.2 Aggregates Treatment

The concentration used to treat the aggregates is 10% for both salts as this is the ideal concentration used in other research cited previously [10, 12]. Portions of the aggregates are soaked in two different solutions of NaHCO₃ and NaCl of 10% concentration by weight. Figure 1 illustrates the protocol followed for the aggregate treatment process. This protocol is divided into two sequences. The first sequence is to study the soaking time of the aggregates in the salt solutions. The second process is to study the influence of rinsing time after each day of treatment. First, the salts are dissolved in preheated water at a temperature between 25 and 30 °C since this is the optimal temperature to dissolve the salts. Then, the prepared samples are added and stirred regularly. Portions of 10 samples per aggregate are taken every 24 h to reach a total duration of 120 h. The extracted samples are dried at 60 °C. These aggregates are then totally immersed for 5 min to measure the pH of the solution and to check the presence or disappearance of salt. Then, these same aggregates are put back in the oven for a new immersion cycle until the salt disappears. They will then be exposed to a wetting and drying cycle to test the efficiency of the treatment.

Microscopic analysis is performed at the beginning of the experiment in the dry condition and was continued through both sequences to assess the dimensional change of the vegetable aggregates. Ten samples are taken per aggregate per treatment day for analysis. These aggregates are measured before the treatment, after the treatment, during each immersion and drying cycle and finally after wetting and drying cycles.

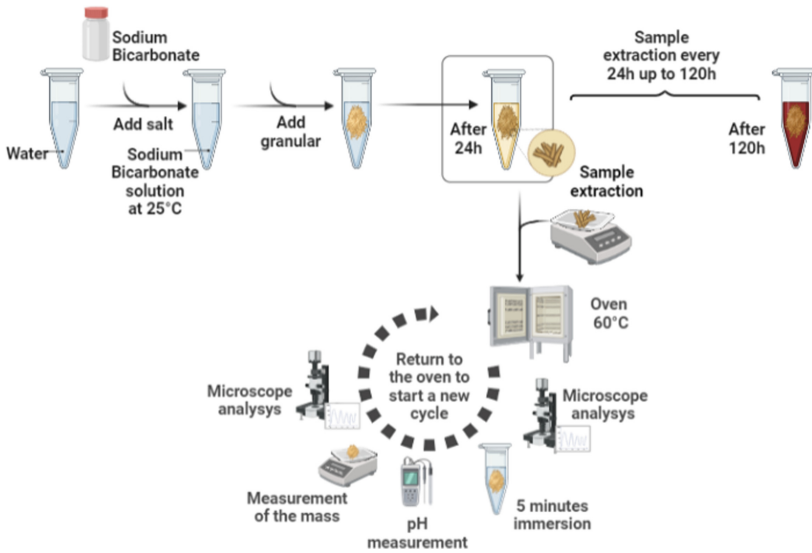


Fig. 1. Treatment protocol.

2.3 Efficiency of Treatment: Wetting-Drying Cycles

The swelling and shrinkage of aggregates due to wetting and drying cycles is observed under a microscope to assess whether the behavior remains constant after several cycles and whether the treatment maintains its effectiveness. This test is performed by subjecting the aggregates to wetting and drying cycles, during which they are wetted at 90% relative humidity at 23 °C and dried at 60 °C with 0% relative humidity. The steps of these cycles are shown in Fig. 2.

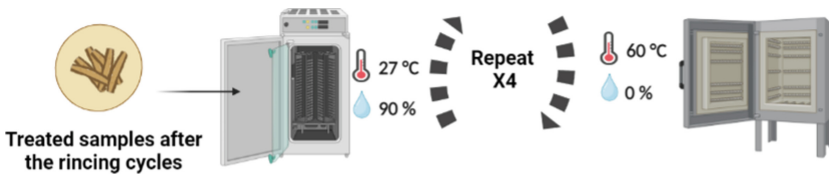


Fig. 2. Repetitive wetting and drying cycle principle

2.4 Microscopic Measurement

The influence of the variation of the treatment time on the morphology of the treated aggregates compared to the untreated aggregates is examined using an optical microscope. An image analysis of the aggregates is carried out using photos taken with the optical microscope “Leica Microsystems (Schweiz) AG”. These images allow to examine the influence of the above processes on the morphology of the treated aggregates compared to the untreated ones. Figure 3 shows the microstructure of each type of aggregate and demonstrates the significant porosity that causes their highly hygroscopic behavior.

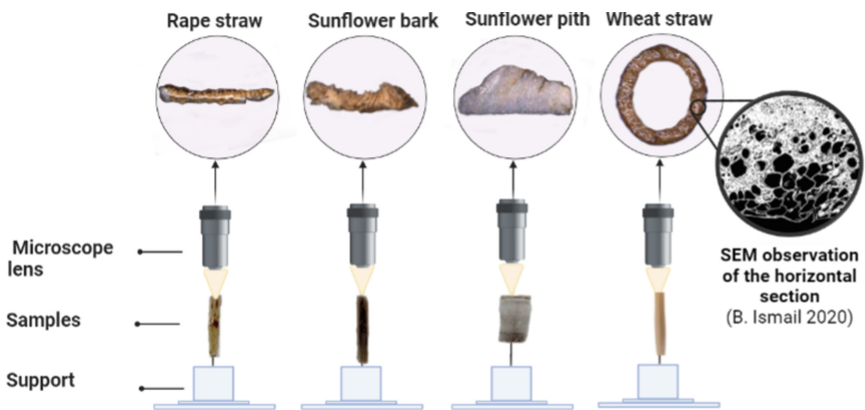


Fig. 3. Different types of aggregates studied as seen via the microscope a) wheat straw, b) rape straw, c) sunflower bark and d) sunflower pith

The microscopic measurement is based on surface measurements. It consists in delimiting the contour of the aggregate and measuring the surface of the aggregate by a region-based segmentation as presented in Fig. 4. This method consists in using the properties of the surface to group the pixels of similar appearance different from the black background. The percentage of variation between two states is defined by the ratio of the variation between the two surfaces to the initial surface. Swelling is thus defined by a positive percentage while a negative sign indicates shrinkage. The measurement error is limited by the distribution of the contour on the aggregate which can be result

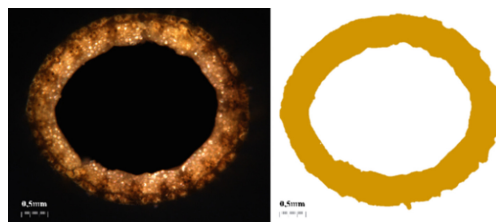


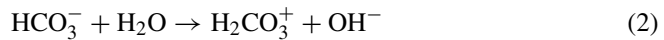
Fig. 4. Delimiting the contour of the aggregates for surface measurements.

from the number of pixels measured, the color and shape of the captured image with a margin of $\pm 2\%$ per measurement.

3 Results and Discussion

3.1 Impact of Treatment

The aqueous sodium bicarbonate solution produces a slightly alkaline solution due to the formation of sodium and hydroxide ions, as shown in Eq. (1) and (2) from which the basicity of the solution over time. The fiber containing hydroxyl groups and Na^+ ions react as Eq. (3).



The amount of Na^+ ions depend on the concentration and basicity of the solutions represented by the pH values. The histograms in Figs. 5, 6 and 7 presents the pH variations of the aggregates treated by NaHCO_3 , NaCl and of untreated aggregates.

Record indicates an increase in basicity of the samples over time to reach a pH of 9 for the NaHCO_3 case. In contrast to the samples that are treated with NaCl , which showed no regular increase but a decrease over days. Nevertheless, untreated aggregates showed an initial variation defined by a decrease during the first 2 days followed by an increase of pH. One of the causes of the decrease in pH in aggregates when water is added can be attributed to the presence of microorganisms within the aggregates [21]. The measurements are repeated four to five times to reduce the error of the measurement to be less than $\pm 4\%$.

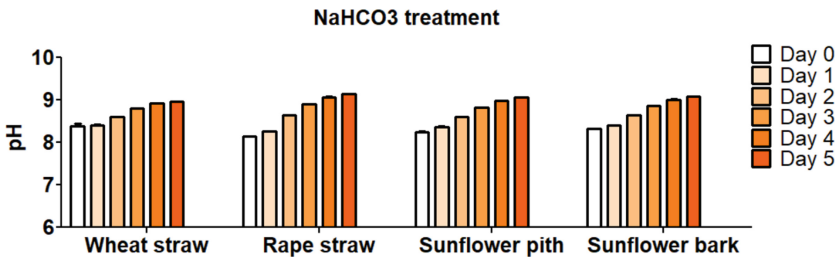


Fig. 5. pH measurement of different aggregates treated with NaHCO_3 solution over days

Treating the aggregates has also a consequence on water absorption since it can causes the change of the chemical composition. Water absorption behavior of wheat straw after treatment is highlighted in Fig. 8 allowing to further define the number of days of treatment. For a better understanding of the water absorption behavior, histograms

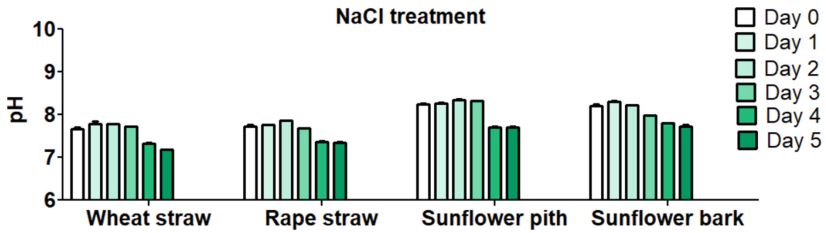


Fig. 6. pH measurement of different aggregates treated with NaCl solution over days

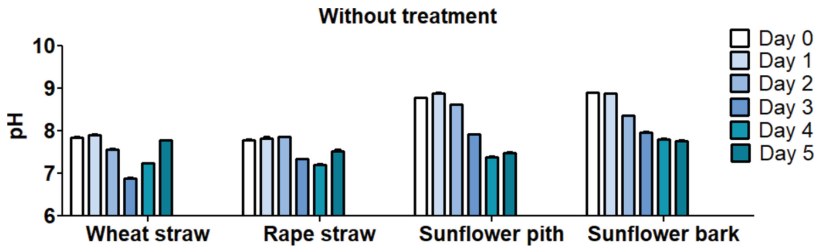


Fig. 7. pH measurement of different untreated aggregates over days.

are presented with the percentage of weight gain with respect to the treatment days. The decrease in water absorption of the treated aggregates is more significant for the aggregates treated with NaHCO_3 rather than for NaCl.

The treatment with NaHCO_3 reduces the water absorption of the aggregates per day of treatment, making them more resistant to moisture. This reduction is mainly due to the change in aggregate composition by the NaHCO_3 treatment. The treatment increases the cellulose content by the partial elimination of hemicellulose [22] which is highly hydrophilic controversy of lignin which is hydrophobic. From the third day of treatment onwards, the moisture absorption becomes 3% and decreases with the day of treatment up to 2.6% for 120 h. Meanwhile, NaCl treated wheat straw remains the same, around 5.5%.

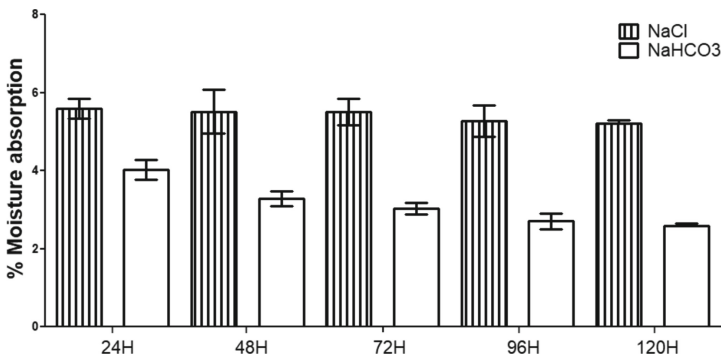


Fig. 8. Moisture absorption as a function treatment time for wheat straw aggregates

Figure 9 illustrates the variations in measurements of the dimensions of the extracted portions before and after treatment. Aggregates treated with NaHCO_3 show a reduction in surface of the treated aggregates from the first day of treatment. After 72 h of treatment, NaHCO_3 solution begins to have a high effect on dimensional variation. As the treatment goes on by day, the surface variation continues to decrease until it reaches a reduction in size of 9% at 120 h.

This agrees with the results of Raharjo et al. [23] who claimed that over the third day of treatment with sodium bicarbonate, the chemical composition of aggregates such as cellulose, lignin and hemicellulose, is modified. Meanwhile, according to Chaitanya et al., beyond 72 h of treatment, the tensile and flexural strength marginally decline. However, excessive removal of hemicellulose beyond 72 h of treatment time leads to a decrease in the load carrying capability of the aggregates.

According to Pejic et al. [24], lignin has an impact on the water sorption of hemp, and it has been shown that increasing the lignin content leads to a decrease in the water retention capacity of hemp fibers. Garat et al. [25] also found that higher levels of lignin are correlated with a lower percentage of swelling. This will strengthen the surface of the aggregates and make it rougher [26]. It can be concluded that after 72 h of treatment, the mechanical properties of the aggregates are enhanced, as indicated by other authors [11] and [9]. The aggregates exhibit a decrease in swelling and shrinkage behavior according to our results. However, NaCl treated aggregates does not prove any dimensional decrease, but a slight increase of for most of them around 2%.

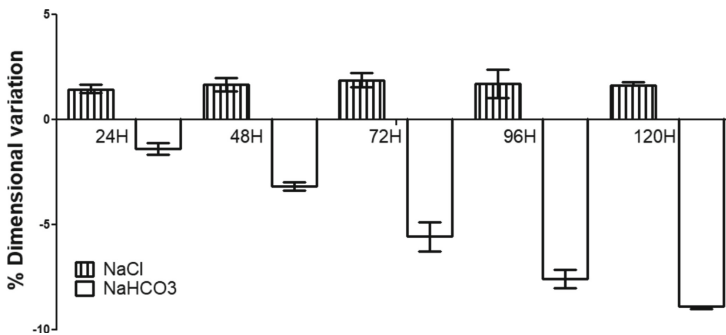


Fig. 9. Difference in measurement between the initial state before treatment and after treatment of the extracted portions per day.

3.2 Rinsing Impact

During the treatment process, samples are extracted and dried in the oven. Then, the rinsing and drying process begins. The aim is to determine the recommended rinsing time to remove excess salt from the aggregate, and to determine how time it takes for the pH to become stable. Figure 10 and 11, present the pH of the aggregates rinsed after extraction during a duration of 72 h of treatment. These aggregates are immersed and dried until they have a stable pH value. The pH of the aggregate treated with NaHCO_3 was 9.2 during

the treatment stage. After the aggregates are dried, the pH of the aggregates increases to 10.2 after the first rinsing. Then, the pH decreases with each subsequent rinsing until it reaches a stable value of 7.8. This variation is also observed in the extracted samples treated for 24 h, up to 120 h. Indeed, when the treated aggregates are dried in the oven at a temperature of 50 °C, the salt of the sodium bicarbonate decomposes under the effect of heat with loss of CO₂ and H₂O. In fact, according to [9], this occurs more normally with high basic pH. The authors [8] found the formation of amine bicarbonate salts in the drying process after treating their aggregate. It is believed that sodium bicarbonate with water creates carbonic acid and hydroxyl component which can react with hydroxyl functional group of flax fiber as similar to alkali treatment and thereby increase the tensile and interfacial properties of composites [27, 28].

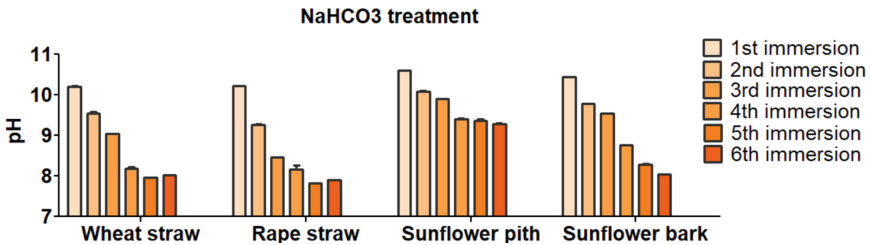


Fig. 10. pH measurement of NaHCO₃ treated aggregates after several rinses.

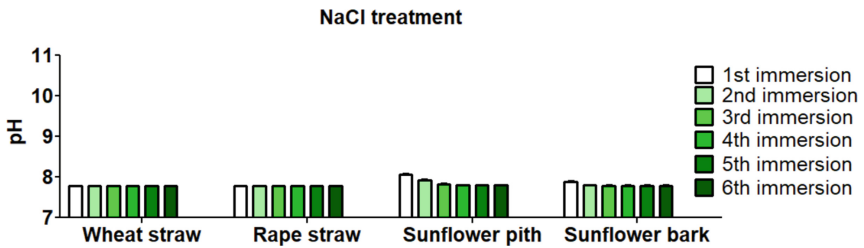


Fig. 11. pH measurement of NaCl treated aggregates after several rinses.

At the same time, the drying of the aggregates at moderate temperature creates a reaction of the amine groups of the sodium bicarbonate with the carbonyl groups of the cellulose to form schiff bases, which is responsible for the yellowing of the material [9]. Indeed, this is what the aggregates treated with sodium bicarbonate has experienced in Fig. 12. The yellowing marked much more the wheat straw, rapeseed straw, than the bark and pith of sunflower. This observation is different from those of treated with NaCl.

After the aggregates have been treated and dried in the oven, they are rinsed several times for 5 min each. The water in which the aggregates were rinsed became lighter and more transparent after the fourth rinse as shown in Fig. 13. The color change is visible when comparing the first rinse (on the left) with the water after the fifth rinse (on the right). Once the aggregates have been rinsed for a total of 20 min, the treatment process is complete, and the aggregates are ready to be used.



Fig. 12. Color change of wheat straw over time: untreated, NaCl-treated, NaHCO₃-treated (left to right).

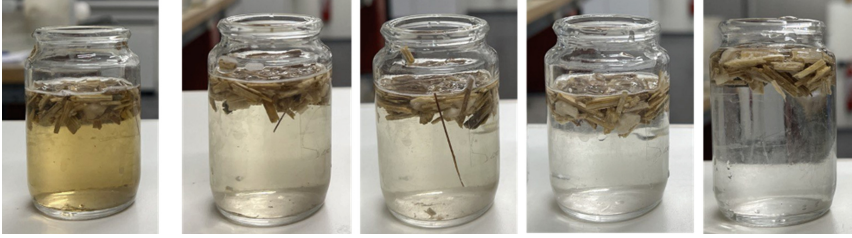


Fig. 13. The rinsing process and color variation of sunflower bark treated aggregates with NaHCO₃ (from left to right).

In summary, the rinsing test identifies the time required to rinse the treated aggregate to remove the amount of salt on the surface. Figure 14 shows the surface of NaHCO₃ treated aggregate after 4 rinses. The amount of salt is quite remarkable after 5 min of rinsing and gradually decreases after the fourth rinsing, for a total of 20 min.

According to the results, it took 20 min of rinsing to remove the salt from NaHCO₃ treated aggregate, while only 5 min of rinsing was needed for NaCl treated aggregate. Therefore, the treatment was repeated using 5 min of rinsing for NaCl treated aggregate and 20 min for NaHCO₃ treated aggregate. Microscopic images (Figs. 15a and c before rinsing the aggregates, 15b and d after rinsing the aggregates) show that the salt was completely removed after the specified rinsing times for NaCl treated aggregates, 5 min, and for NaHCO₃ treated aggregates, 20 min.

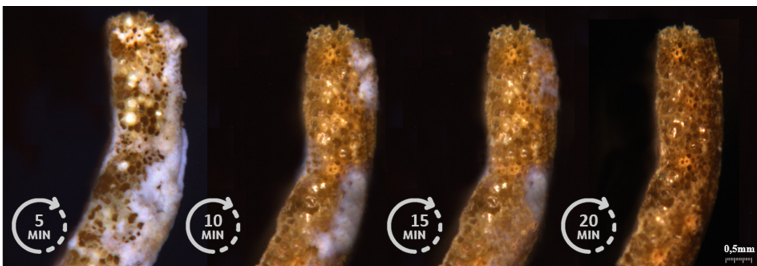


Fig. 14. Salt disappearance during time for nahco3 treated aggregates.

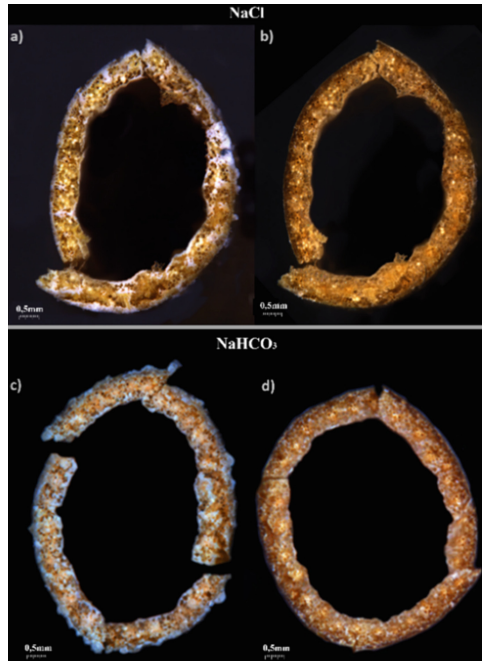


Fig. 15. Surfaces of treated aggregates: a) and c) before rinsing and b) after 5 min of rinsing for NaCl and d) after 20 min of rinsing for NaHCO₃.

3.3 Wetting and Drying Cycles

The percentage of dimensional change in the aggregates after wetting and drying cycles is shown in Fig. 16. Treatment with Sodium bicarbonate (NaHCO₃) leads to a significant decrease in swelling by 60% ± 3%. This can be seen by comparing the swelling of untreated aggregates (21%) to that of aggregates treated with NaHCO₃ (8%). Based on the data, the NaHCO₃ treatment is more effective and durable than NaCl treatment, as it does not show a decrease in effectiveness over time. The untreated samples began to become extremely soft, which impacted the swelling measurements.

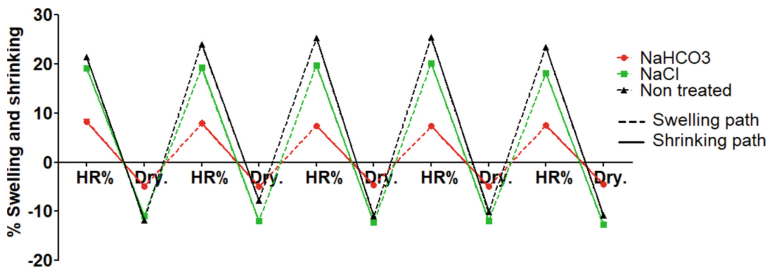


Fig. 16. Humidification and drying cycle for wheat straw for NaHCO₃ treated aggregates, NaCl treated aggregates and non-treated aggregates.

4 Conclusion

The study of the effectiveness of the treatment protocol of plant aggregates and the treatment effect on swelling and shrinkage is investigated. It is important to adapt a treatment protocol on vegetable aggregates that will have an influence on the bio-composite. According to the results obtained from pH measurements, microscopic analysis and weight analysis, the adaptation of the treatment for 3 days, followed by a drying at moderate temperature at 50 °C and then a rinsing of 20 min is recommended for NaHCO₃ treatment while only 5 min of rinsing for NaCl treatment.

For aggregates treated with 10% sodium carbonate, there is a notable reduction of swelling and shrinkage by $60 \pm 3\%$ in comparison to untreated aggregates. However, the NaCl-treated aggregates did not show a high enough rate of swelling and shrinkage prevention. A remarkable improvement of sodium bicarbonate treated aggregates to decrease their dimension variation is related to the removal of hemicellulose from the interfibrillar region according to the literature. It reduces water absorption up to 2.6% for 120 h and consequently swelling and shrinkage up to 10%.

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