Evaluating Effects of Activated Sludge and Nanochitosan on Physical and Strength Properties of Recycled Pulp

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The present research aims to shed light on the effect of activated sludge (from a paper mill) and nanochitosan on the physical and strength properties of recycled pulp. Firstly, activated sludge was treated with 3% acetic acid for 30 min and then placed in a beaker for 90 min at 100 °C. Then, the ingredients were mixed and refined with recycled newsprint pulp in different proportions (0, 5, 10, and 15%). Finally, 2% nanochitosan was optionally added. Test specimens were prepared according to TAPPI standards with a basis weight of 120 g/m^2, and their physical (water absorption) and strength (tear strength, tensile strength, burst strength, and ring crush test) properties were measured and compared. The results showed that with the increase of untreated activated sludge in recycled paper pulp, the indicators of tear resistance, ring crush test, and burst strength decreased and water absorption increased. Strength properties increased and water adsorption decreased when adding activated sludge treated with 3% acetic acid. Through the addition of nanochitosan to activated sludge treated with acetic acid, a significant increase in strength properties and a decrease in water absorption were observed.

Keywords: Acetic acid; Waste-paper; Activated sludge; Nanochitosan

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INTRODUCTION

In recent years, population explosion and rising living standards have led to higher consumption and production of paper along with other industrial and agricultural products, and as a result, the amount and variety of solid waste produced has risen accordingly. Environmental pollution is making agricultural and industrial waste a global concern (Soucy *et al*. 2014). Therefore, finding appropriate social, technical, economic, and environmental solutions to achieve a greener and cleaner environment is important (Pappu *et al*. 2007; Lertsutthiwonget *et al*. 2008). Mazandaran Wood and Paper Industries Complex (Mazandaran, Iran) is designed for annual production of about 75,000 tons of testliner and fluting board, 52,000 tons of newsprint, and 38,000 tons of printing paper (Ghafari *et al*. 2019). In this factory, a considerable amount of sludge from rapid oxygenation of wastewater in secondary clarifiers are produced, called activated sludge. The average activated sludge (organic and inorganic) produced in this plant is approximately 1700 tons per month, of which the dry matter percentage is approximately 25%. About 14 tons of completely dry sludge is produced daily in this factory, which is practically unused and is currently being buried (Son *et al*. 2001). The landfill of paper mill waste is not being economical, and it causes groundwater pollution. Furthermore, a large area of land is required for the landfilling of fibers (Cavdar *et al*. 2017). Due to the lack of primary wood resources, the growing demand for paper and paper products, and environmental issues, the pulp and paper industry needs to recycle waste and waste paper (Smook 1995; Moo-young and Ochola 1999).

Recycling processes, despite the advantages and potential intrinsic properties, are often associated with a sharp decline in product quality and strength properties (Mandeep *et al*. 2020). Therefore, in the recycling process to improve the bonding between the fibers and increase the strength properties of products, the development of various methods, including further refinement, addition of virgin fibers, and use of cationic additives, use of industrial wastes (activated sludge of paper mill), along with common and developing additives to improve the strength and properties of paper is common (Rudi 2018). Chitin is the second most abundant natural biopolymer after cellulose. It is structurally similar to cellulose, except that chitin has acetamide ($-NHCOCH_3$) groups in the carbon C2 position. The similarity of chitosan to cellulose has led to its good compatibility with cellulose pulp fibers (Rahmaninia *et al*. 2015). Additionally, the amino polysaccharide chitosan is an excellent linker for cellulosic fiber structures and can be up to 40% more efficient than starch (Vanerek *et al.* 2006).

Chitin and chitosan and their derivatives have a wide range of applications in paper production. They can be used to solve various problems in wastewater treatment as well as improve the efficiency and effectiveness of paper machines, improve paper strength, and prepare packaging materials. In general, given the biodegradable nature of chitosan, it is obvious that chitosan can be a viable alternative to the production of environmentally friendly papers (Song *et al*. 2018). Sabazoodkhiz *et al*. (2017) in the study of the interaction of chitosan biopolymer with silica nanoparticles as a strengthening agent and preservative in recycled cellulose fibers, showed that this system improves the drainage process and preserves fillers and fibers.

Considering the environmental and economic dimensions, 1% chitosan and 0.1% nanosilica were well treated with 42 and 168% increases in drainage and filler retention, respectively, compared to the control sample. Improvement of mechanical and physical properties, such as density, specific gravity, tensile index, and tear, indicates the proper performance of this treatment.

Girones *et al.* (2010) stated that increasing the amount of sludge in PP-composites will result in a PP-composite with lower tensile strength. Therefore, in PP-composites that are exposed to tensile force the paper mill sludge acts as a filler, and when the material is subjected to bending force, the sludge acts somewhat like a reinforcement. Ismail and Bakar (2004) reported that increasing the amount of paper sludge in composites increases water absorption and decreases tensile strength. Ismail and Bakar (2005) reported that the ester treatment and acetylation of the sludge of the paper mill in the manufacture of composites improves the tensile strength and Young's modulus and also reduces water absorption.

The aim of this research was to use activated sludge as an additive and also to use modified activated sludge with acetic acid and add nano-chitosan to make more functional groups available on the surface of treated activated sludge to increase bond strength and improve the physical-mechanical properties of paper produced from recycled fibers.

EXPERIMENTAL

Materials

In this study, 10 kg of waste newspaper with dimensions of 5 cm × 5 cm was prepared. Dry activated sludge was prepared from Mazandaran Wood and Paper Factory located in Kola area of Sari, Iran and transferred to a specialized wood and paper laboratory. After keeping in water for 24 h, the newspaper pieces were pulverized for 30 min at a speed of 300 rpm using a disintegrator (British Pulp Evaluation Apparatus manufactured by Mavis Engineering Ltd., London, England). Then, approximately 2 kg of activated sludge was soaked in water for 24 h. Activated sludge had a pH of 7.1, a dry matter percentage of 55.65, an organic matter content of 55.2%, and a mineral content of 44.8%. After that, newspaper pieces and activated sludge were mixed with a disintegrator at 300 rpm. Then, the newspaper pulp was dewatered with 40-mesh and sludge with 200-mesh. The waste newspaper pulp and activated sludge were calculated and mixed in the amount required for the experiment according to Table 1. Waste newspaper pulp and activated sludge were refined using a 300 rpm PFI-mill refiner (No. 221; Beating Performance Adjusted by the Norwegian Pulp and Paper Research Institute, Trondheim, Norway). Test samples in 4 levels were prepared according to the TAPPI T205 sp-12 (2015) standard with a basis weight of 120 g/m^2 (Table 1).

Table 1. Ratio of Sludge to Waste-paper in Making Test Samples

Waste-paper (%)	Sludge (%)	Row
100	0	1
95	5	2
90	10	3
85	15	4

Table 2. Mineral Compounds of Activated Sludge

Titanium Oxide (%)	Manganese Oxide (%)	Phosphorus Trioxide (%)	Potassium Oxide (%)	Sodium Oxide (%)	Aluminum Oxide (%)	Magnesium Oxide (%)	Silica (%)	Calcium Oxide (%)
0.002	0.04	0.3	0.3	0.3	4.7	10.6	13.7	6.68

According to Table 2, the highest and lowest amounts of mineral compounds were related to silica and titanium oxide, respectively.

Chemical treatment of activated sludge with acetic acid

To improve the mixing conditions with newspaper pulp and activated sludge, the sludge specimens with the ratios listed in Table 1 were placed separately in beakers containing 3% acetic acid for 30 min. Then, they were treated in a Bain Marie bath (90 min and 100 °C). Test specimens were prepared and pressed according to the TAPPI T205 sp-12 (2015) standard with a basis weight of 120 g/m^2 and then dried. Finally, the strength and water absorption properties of activated sludge and waste-paper pulp were measured. In the next step, activated sludge was treated again with acetic acid under conditions (90 min and 100 °C), and 2% nanochitosan was added to this suspension.

Test samples were prepared and pressed according to TAPPI T205 sp-12 (2015) standard with a basis weight of 120 g/m^2 and then dried. Finally, the resistance properties and water absorption of the samples were measured.

Nanochitosan preparation

Chitosan is a cream-colored, clear powder made by Seafresh, Bangkok, Thailand, with a molecular weight of 270 Da, an acetylation grade of 93%, and an average diameter of 55 nm. To inject the chitosan solution into the pulp slurry, the required amount of chitosan was dissolved in 1% acetic acid. For this purpose, the mentioned solution was stirred for 2 h at room temperature. When making a laboratory sample, first 2% chitosan polymer was added to the turbulent pulp with a rotational intensity of 500 to 300 rpm, (Nicu *et al.* 2011).

Methods

Measurement of physical and resistance properties of test specimens

Tensile strength, burst resistance, tear strength, ring strength, and water absorption indices were tested in accordance with TAPPI T494 om-13 (2015), TAPPI T403 om-10 (2015), and TAPPI T496 sp- (2015) standards, TAPPI T818 cm-07 (2015) and TAPPI T441 om-13 (2015), respectively.

ATR and FTIR spectroscopy

Fourier transform infrared (FTIR) spectroscopy was used to determine the status of the active groups present in the handheld samples made from untreated and acetic acid-treated paper pulp and sludge. For this purpose, the JASCO FTIR-4700 device (Japan Spectroscopic Co., Ltd., Hachioji City, Tokyo, Japan) was used.

Electron microscopy evaluation

The SEM device used under the brand MIRA3 FEG-SEM was made by Tescan Company in Brno, Czech Republic. This device had a field emission film and was applied with a voltage of 10 to 15 kW.

Statistical analysis

In this study, the effect of activated sludge on physical and strength properties of recycled pulp obtained from mixing activated sludge and newspaper waste-paper was statistically evaluated.

Statistical analysis was performed using SPSS software (IBM Corporation, v.23, Armonk, NY, USA) in the form of one-way analysis of variance. Finally, the means were grouped using Duncan's test at a confidence level of 5%.

RESULTS AND DISCUSSION

Attenuated Total Reflection (ATR) Spectroscopy

The spectrum (ATR) in control (0% sludge), untreated, and acetic acid-treated sludge is shown in Fig. 1.

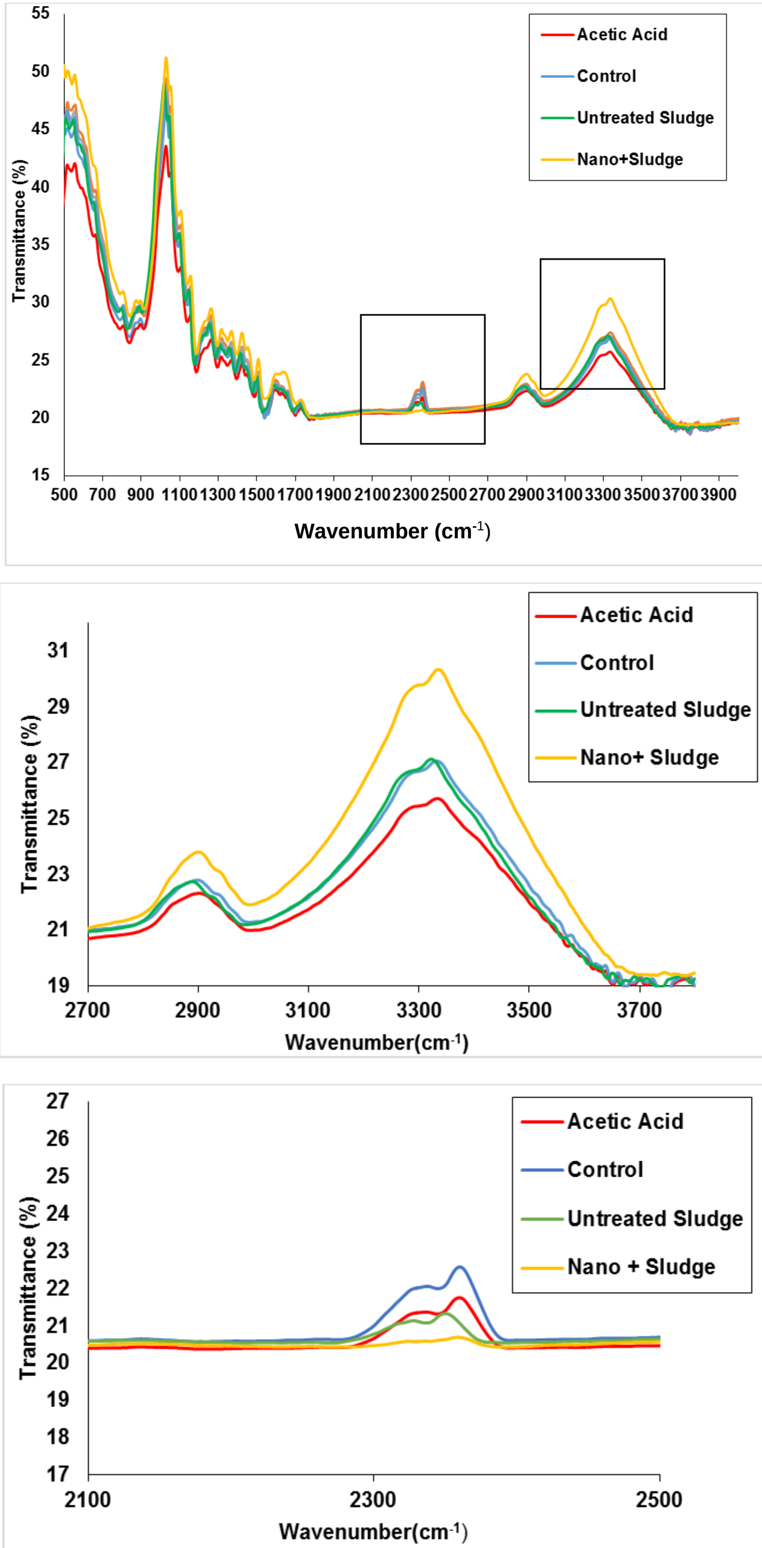

Fig. 1. ATR diagram and its insets

Mechanical and Physical Properties

Mechanical properties

The results of analysis of variance showed that there was a significant difference in the strength properties of the paper samples ($P < 0.05$). The energy consumption during tearing of the paper was reduced by the addition of untreated sludge (Fig. 3). Comparing the results of untreated activated sludge and the control papers indicates less resistance of paper made with untreated activated sludge. Significant changes after modification in different bands were attributed to different components, which include cellulose, hemicellulose, and lignin. The decrease in spectrum intensity at 3300 cm^{-1} wavelength due to treatment can be attributed to hydroxyl (OH) groups (Sabazoodkhiz *et al.* 2017). The hydroxyl tensile region is of particular importance for explaining hydrogen bonding patterns (Clausen *et al.* 2010) (Fig. 1). Bagherzadeh (2013) stated that the decrease in strength is reduced with increasing the filler surface area, because the filler materials in the paper act as a separator, reducing the fiber connections and thus reducing the tear strength (Bagherzadeh 2013). Barzan *et al*. (2015) also reported that compared to control treatment paper, handmade papers filled with extracted calcium carbonate reduce tear resistance by approximately 19%. Tear resistance index in papers made with activated sludge treated with acetic acid showed a significant increase compared to untreated. In untreated activated sludge, dislocation, fiber rupture, and fiber breakage occurred (Fig. 2A). However, after acetic acid treatment, more uniform surfaces and less fiber breakage were seen, which can indicate the improvement of fiber surface by an increase of bonding and better paper formation, and that acetic acid treatment provides a more suitable substrate for the physical and mechanical properties of paper (Fig. 2B). Through adding nanochitosan to acetic acid-treated samples, a significant increase was achieved at all mixing levels (Figs. 3 to 7).

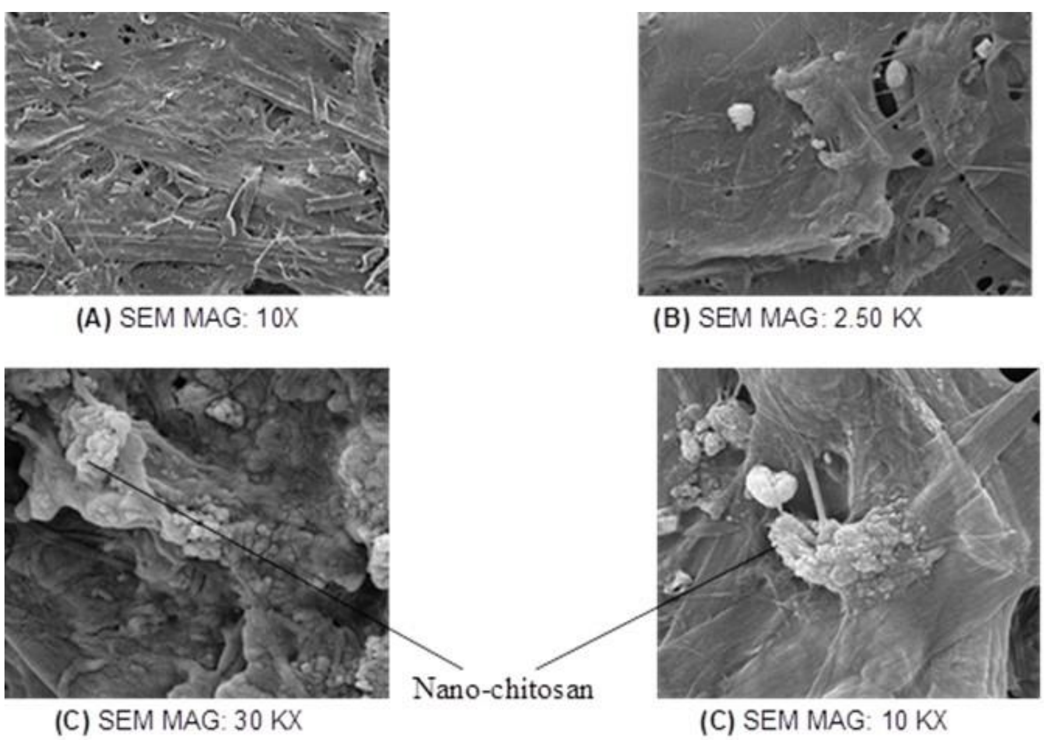

Fig. 2. SEM images of (A) paper from untreated sludge, (B) paper from sludge treated with acetic acid, and (C) paper from sludge treated with acetic acid + nanochitosan

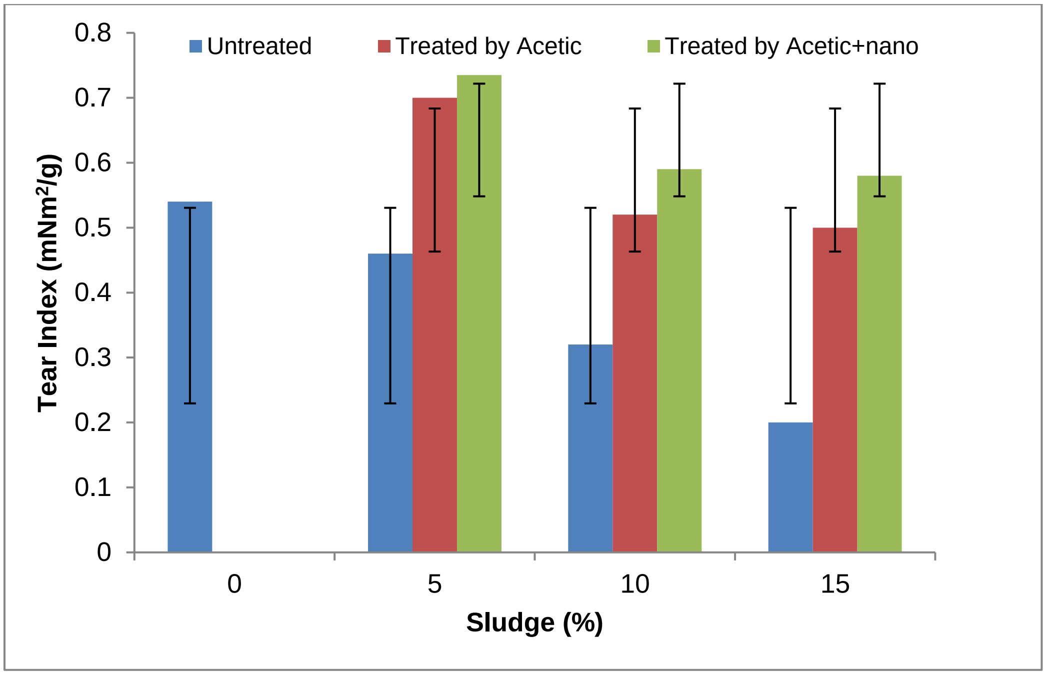

Fig. 3. Comparison of tear strength index of samples obtained by adding activated sludge to waste-paper

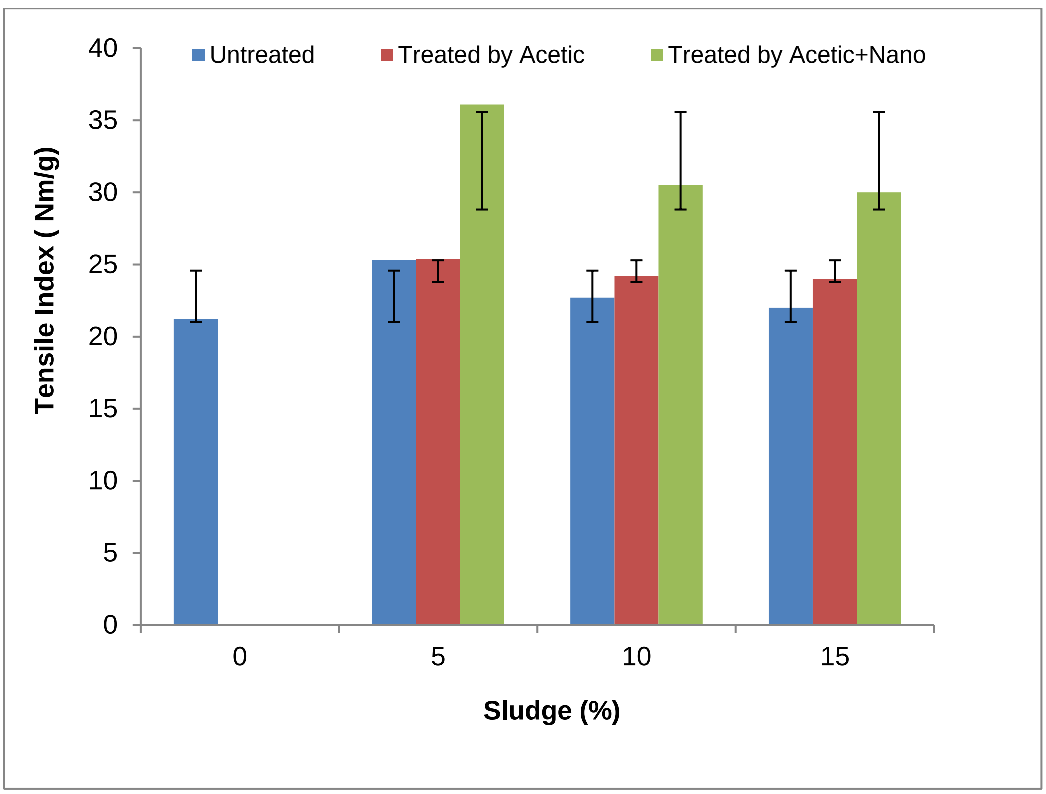

Fig. 4. Comparison of tensile strength index of samples obtained by adding activated sludge to waste-paper

The addition of 5% activated sludge treated with acetic acid and nanochitosan caused the highest tear resistance index efficiency in the samples. The dispersion of nanoparticles after the addition of nanochitosan to activated sludge treated with acetic acid, while treating and modifying the surface of the fibers, caused less agglomeration and also improved the adhesion of fibers by nanochitosan (Fig. 2C). The FTIR spectrum of nanochitosans, in the regions of 1300 and 1700 cm^{-1}, shows the acetyl functional group in the treated samples and the esterification reactions (Neeru *et al.* 2013) (Fig. 1).

The tensile strength index of the samples can be seen in Fig. 4. Tensile strength is one of the most important strengths of paper that protects it against tensile stresses. Paper weight, fiber roughness, and fiber width are the factors affecting tensile strength. Tensile strength increases with increasing fiber length, increasing refining intensity, increasing press pressure, and performing surface sizing on paper (Scott 2007). The results showed that the tensile strength index was increased by adding untreated activated sludge to the recycled fiber pulp. The reason for this increase can be attributed to the high specific surface area of the activated sludge (Nazeri *et al.* 2008). Primary and secondary materials improve the strength properties of the paper (Krogerus *et al*. 2002). Son *et al.* (2004) showed that the swelling and tensile properties of the composites improved slightly with the addition of paper sludge compared to the control samples (Son *et al.* 2004). In contrast, the results of other studies have shown that the treatment of esterification and acetylation improved the tensile strength and length change in the fracture zone as well as the Young's modulus in composites and reduced water absorption (Ismail and Bakar 2005).

The removal of the corresponding peak in the adsorption region of 1248.3 cm^{-1} in activated sludge can be due to the flexural vibrations of phenolic hydroxyl. Additionally, the removal of peaks in the adsorption zones of 1237.0 cm^{-1} is closely related to the C=O, C=C, and C=O bonds of the carbonyl units, and the adsorption region of 1412.1 cm^{-1} is related to aromatic C-H and C-H associated with type II alcohols, as well as C=O is treated in activated sludge and attenuates or removes carbonyl groups (Fig. 1) (Mehmood *et al.* 2019). As a strength additive, chitosan has a structure similar to cellulose filaments, and thus it uses hydrogen and Van der Waals bonds to improve bonding between fibers, thereby improving tensile strength (Li *et al.* 2004). Samples with 5% sludge treated with stick and nanochitosan had the highest tensile strength.

Figure 5 compares the ring crush test of the specimens. The highest values of this index were observed in cases where 15% of sludge was added to waste-paper pulp and in samples treated with stick and nanochitosan.

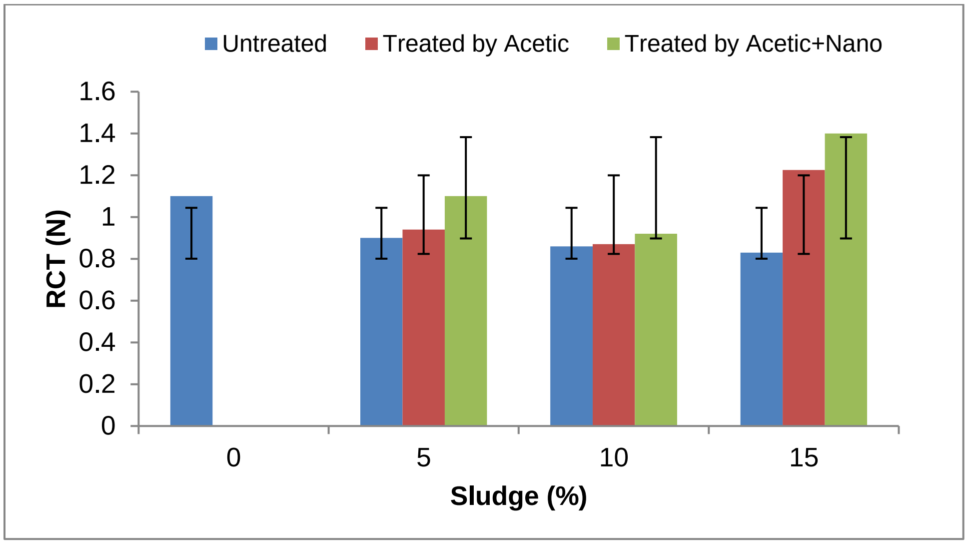

Fig. 5. Comparison of ring crush test (RCT) of samples obtained by adding activated sludge to waste-paper

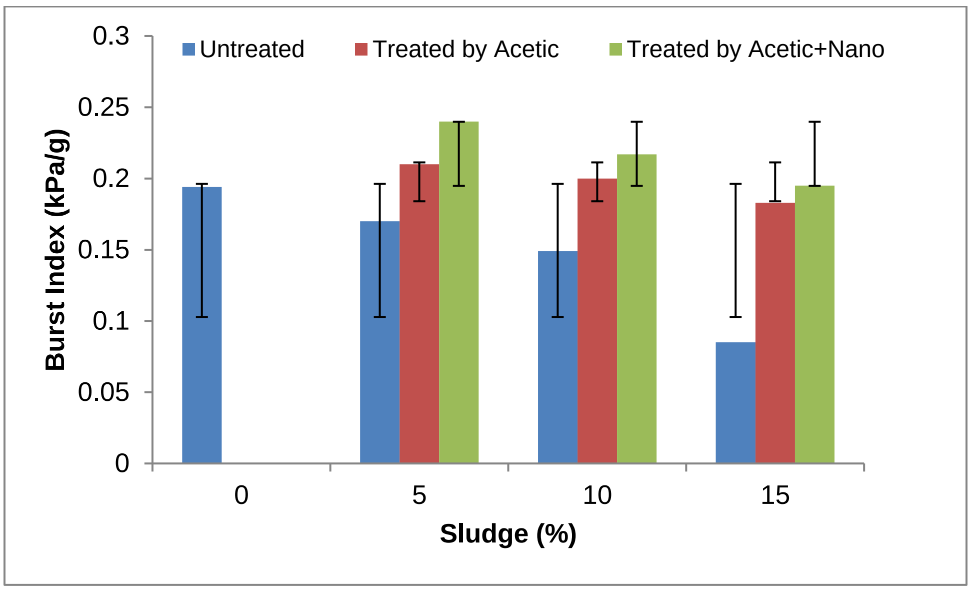

Fig. 6. Comparison of burst resistance index of samples obtained by adding activated sludge to waste-paper

The burst strength index decreased with the addition of untreated activated sludge. This can be due to the presence of more fillers between the fibers, such as calcium carbonate (polymorphism of calcite), and the small particle size of calcium carbonate fillers in untreated activated sludge, which reduces the level of bonds. In contrast, the addition of activated sludge and weakening of the bond between the fibers reduces the mechanical strength (Akbari *et al.* 2017). Additionally, the short fibers length in activated sludge and the use of recycled fibers can be another way to explain the reduced mechanical properties (Miri *et al.* 2016). The highest percentage of burst resistance index was related to the consumption conditions of activated sludge (5%) (Fig. 6). Hemicellulose and lignin are affected by the treatments, which reduces the amount of hydroxyl groups and has a significant effect on lignin (Mehmood *et al.* 2019). Aliphatic groups are seen in the absorption range of 3312 cm^{-1} (Fig. 1). Additionally, samples treated with acetic acid and the addition of nanochitosan had the highest values related to this index.

Physical properties (water absorption)

The results of analysis of variance showed that there was a significant difference in the physical properties (water absorption) of the made paper samples ($p < 0.05$). In papers made of untreated activated sludge, it was observed that with increasing the percentage of activated sludge, the average water absorption increased (Fig. 7). Therefore, hydrophilic structures in activated sludge as well as moisture absorption by recycled newspaper fibers can be a reason to reduce the strength properties and increase water absorption. In contrast, the percentage of water absorption was significantly reduced by adding nanochitosan. Additionally, the region of the 2300 cm^{-1} peak belonged to the carbonyl groups, which indicates hemicellulose leaching (Pandey *et al.* 2012) (Fig. 1).

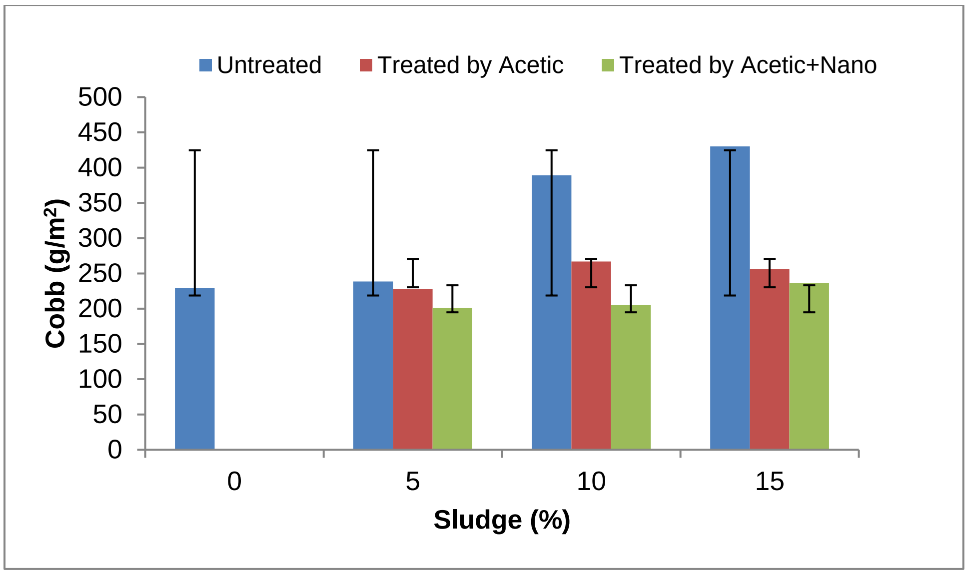

Fig. 7. Comparison of water absorption of samples obtained by adding activated sludge to waste-paper

Ismail and Bakar (2004) reported that increasing the amount of paper sludge in composites increases the Young's modulus and water absorption.

In papers made of activated sludge treated with acetic acid, a decrease in water absorption index was observed by increasing the percentage of activated sludge. Then, by adding nanochitosan to acetic acid-treated samples at all mixing levels, a significant reduction in water uptake was obtained. Decreased water absorption represents a favorable change in physical properties, and the lowest water absorption was observed at the mixing level of 5%. With the simultaneous addition of activated sludge and nanochitosan, a noticeable decrease in the water absorption of the resulting paper was observed, but this decrease in water absorption was more influenced by chitosan. The ability to establish hydrogen bonding between chitosan amino groups and hydroxyl groups of fibers is one of the theories of bonding chitosan with cellulose fiber surface. The possibility of forming electrostatic bonds between anions of surface fibers and activated sludge is another reason. The ability to form a covalent bond through the reaction of chitosan with aldehyde groups of fibers is one of the reasons for the bonding chitosan to the surface of cellulosic fibers, which ultimately leads to a reduction in some water-absorbing groups (Myllytie *et al.* 2009). Nanochitosan, as a biopolymer and a natural biopolymer, has amino groups that allow it to have a cationic charge in acidic conditions. Additionally, its direct polymer chain, which consists of the monomer (1-4) 2-amino-2-deoxy-beta-di-glucan, increases the adhesion between the fibers and increases the strength (Sabazoodkhiz *et al.* 2017).

Increased strength also indicates the effect of nanochitosan. In addition, significant infiltration, effective size, and uniform distribution of nanochitosan and activated sludge can reduce moisture and improve dimensional stability (Rautkari *et al*. 2013). The presence of nanochitosan disrupts the formation and increases the strength level of the paper because chitosan, due to the nature of the positive charges, can disrupt the formation. Acetic acid-treated sludge and the addition of nanochitosan increased the bonding points and resulted in better formation. It seems that the deposition of chitosan on pulp fibers and combined with the appropriate level of carboxyl groups led to good formation. In general, the study of chitosan adsorption behavior on the surface of cellulosic fibers shows that the nanochitosan layer causes more bonding and increases the physical strength of the paper (Schwanninger *et al*. 2004).

CONCLUSIONS

The main purpose of this study was to improve the physical and mechanical properties of paper made from activated sludge and recycled fibers by acetic acid treatment with acetic acid and the addition of nanochitosan.

1. The results showed that increasing untreated activated sludge to waste-paper reduced mechanical strengths (tear strength index, ring-crush test (RCT), burst strength index, and tensile strength index) by approximately 22% and increased water absorption by approximately 35%. Chemical treatment of activated sludge with acetic acid and its addition to recycled pulp caused a significant increase in tear strength index (88.3%), tensile strength index (5.3%), RCT (17.7%), and burst strength index (57.6%) and reduced water absorption (25.3%) compared to untreated activated sludge.
2. Further, chemical treatment of activated sludge with acetic acid and the addition of nanochitosan to waste-paper caused a significant increase in tear strength index (111.4%), tensile strength index (37.8%), RCT (32.6%), and burst strength index (72%) and reduced water absorption (36%) compared to the use of untreated activated sludge.
3. In contrast, the addition of nanochitosan increased the strength properties and reduced the water absorption more than the control sample and activated sludge treated with acetic acid.
4. According to the obtained results, the best mixing percentage for hand-made samples was at the level of 5% of activated sludge treated with acetic acid and the addition of nanochitosan.

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