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Mechanical effects of sodium pentaborate pentahydrate and sodium pentaborate anhydrate additives in polypropylene

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ÖZET

This research examines the effect of micro size sodium pentaborate pentahydrate and anhydrate forms on the physicochemical and mechanical properties of filled polypropylene (isotactic homopolimer – Borealis HE125MO) composite matrices. The Sodium pentaborate pentahydrate and anhydrite dried 105°C 1 hour and mixed with micro compounder with polypropylene (with different proportions) and produced test samples with injection molding method. Mechanical properties were measured (tensile strength, modulus of elasticity, ultimate strength, tensile at yield (at room temperature)) along with hardness (charpy impact) test. Increasing filler content lead to an increase mechanical strength of the composite material depends on the percentage quantity of mixture proportion. The observed increase in tensile strength is %4,2 with %1 sodium pentaborate pentahydrate additive. The modulus of elasticity increases with additive quantity and the ultimate strength and strain at yield effected parallel with tensile strength change. With high proportion (%5-%10) of filler content, the mechanical properties decreased but increased the hardness. To understand the physicochemical effects, FTIR test applied on samples and O-H bonds effects and C-C bonds change observed. According to Infrared spectroscopy, the polypropylene had changed 1,56% with sodium pentaborate pentahydrate additive and 2,86% with %1,5 additive of sodium pentaborate anhydrite form. The fingerprint region alterations at FTIR tests and water effect observed. The molecular bonding effects of sodium pentaborate with chain of isotactic homopolimer examined. The nucleating effect of sodium pentaborate in polypropylene is viewed with SEM test. In microstructure, positioning effects of sodium pentaborate particles in chain/fiber structure of polypropylene observed and associated with mechanical properties and physicochemical impacts.

I. INTRODUCTION

In the past two decades, polymeric materials have become critically important due to their high usage volumes and production capabilities [1]. Consequently, polymer materials are favored for numerous engineering applications. This is particularly true in the white goods and automotive industries, where the processing of products necessitates the use of polypropylene, polyethylene, polycarbonate, polystyrene, and polyamide components in their exterior and other functional parts [2].

The mechanical properties of plastic materials make the design of injection-molded plastic parts quite challenging, prompting manufacturers to seek efficiently developed plastic materials for better design solutions. Mineral additive systems, especially semi-crystalline structures, are among the most preferred for achieving good mechanical properties and cost-reducing design solutions [3]. One of the most used polymeric materials in industries is polypropylene, favored for its semi-crystalline microstructure and the mechanical properties that can be enhanced with mineral additive systems. This material provides significant advantages in terms of low density, high chemical resistance, thermoplastic processability, and cost-effectiveness [4].

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On the other hand, there exists a mineral that requires a polyamide additive to enhance surface tension [4]. Sodium pentaborate (SPB) is a rare natural mineral composed of boron, sodium, oxygen, and hydrogen. Sodium pentaborate is utilized in various industrial applications. Sodium pentaborate pentahydrate (NaB) and Hypericum perforatum (HP) oil can be integrated into a polyvinyl alcohol (PVA) and chitosan (CH) polymer matrix for use in wound healing [5]. Furthermore, sodium pentaborate can be added to a NaAlO2 + KOH electrolyte PEO coating to provide corrosion protection [6]. Moreover, the combination of sodium pentaborate pentahydrate and cisplatin (Cis) materials can produce effects against cancer cells, inhibiting cell proliferation [7]. In addition to its mineral properties, sodium pentaborate exists in two distinct forms; one is known as pentahydrate (SPP) [NaB5O8-10H2O], which contains water, while the other is referred to as anhydrite (SPA) [NaB5O8], which is devoid of water.

Sodium pentaborate ($NaB_5O_8 \cdot 5H_2O$) is a notable salt among boron compounds, which has gained increasing significance in various industrial sectors in recent years. This compound, particularly utilized as an additive in ceramics, glass, polymer composites, and metal coating technologies, is favored due to its advantages such as low toxicity, high thermal stability, and ion exchange capacity. Recent studies have revealed the potential of sodium pentaborate to enhance mechanical surface properties [8]. Similarly, sodium pentaborate functions as a friction reducer, pH stabilizer, and corrosion inhibitor in liquid systems used for oil and natural gas drilling, thereby improving the mechanical strength and efficiency of drilling equipment [9]. These versatile applications highlight sodium pentaborate as a strategic compound in advanced material technologies, both as a structural and chemical additive.

SPB, as a component of enzymatic liquid detergents, can be utilized as a feed additive to prevent viral and bacterial pathogen contamination in shrimp, and serves as a pharmaceutical component to enhance skin wound healing rates [10-12]. It is known that ceramic additives have positive mechanical effects on polypropylene and organic materials such as talc, calcite, as well as inorganic materials like activated carbon and fiberglass [2, 13-16]. For instance, the integration of SPB nanoparticles into the AZ31 magnesium alloy has been studied for corrosion effects, resulting in significant improvements on the obtained surface [17]. Furthermore, the sodium pentaborate compound can be incorporated with eggshell (CaCO3) and natural resin additives to produce a biofilm that protects against UV rays, making it applicable in packaging production [3]. Additionally, the interaction of sodium pentaborate with various substances can also be utilized in the development of antimicrobial products [18].

The Fourier Transform Infrared Spectroscopy (FTIR) method is employed to observe and determine the chemical structure of polymers. For example, structures created by the addition of colemanite to thermoplastic polyurethane (TPU) have been analyzed for their chemical structure using the FTIR method, by combining sodium pentaborate pentahydrate (NaB) and hypericum perforatum (HP) oil solution casting method with a mixture of polyvinyl alcohol (PVA) and chitosan (CH) polymers to obtain membranes [1, 6].

Sodium pentaborate crystals promote the growth of crystals by inducing supersaturation when deionized water is utilized. To ascertain the thermal and mechanical properties of these growing crystals, TG-DTA and Vickers hardness tests are conducted [19]. Furthermore, to comprehend the thermal and mechanical properties of ulexite, boric acid-borax mixtures, and zinc borate fillers, thermogravimetric analysis, tensile tests, and three-point bending tests have been employed to evaluate their effects [15, 20, 21]. Additionally, the composite structure formed by sodium pentaborate and the AZ31 magnesium alloy can be visualized using scanning electron microscopy (SEM) to examine its phase structure [17]. To investigate the impact of sodium pentaborate (B5H10NaO13) in the PEO coating on the WE43 magnesium alloy, SEM is utilized alongside ball wear tests, scratch tests, and potentiodynamic corrosion tests [7].

In this study, the mechanical effects of sodium pentaborate pentahydrate and sodium pentaborate anhydrous additives on polypropylene have been examined. The polypropylene material was specifically selected as isotactic, and composites produced through specialized contamination were utilized to understand the effects. Literature and testing techniques, including tensile strength tests, FTIR (Fourier Transform Infrared Spectroscopy), and SEM (Scanning Electron Microscopy), were employed to identify microstructural changes.

Through this research, the production methods of polypropylene with SPA and SPP additives were analyzed, focusing on properties such as elasticity moduli, yield strength, and surface tension, as well as the mechanical effects of SPP and SPA in polypropylene. An FTIR test was conducted to comprehend the microstructure and intermolecular bonds, and the results obtained were evaluated. The relevance of molecular bonds on polypropylene was investigated. SEM illustrated the micro chain structures of polypropylene and the positions of SPB and SPA.

II. EXPERIMENTAL METHOD

2.1 Materials

The isotactic polypropylene (iPP) which is Borealis HE125MO homopolimer intended for injection molding [19]. This grade is characterized by good flow properties and high stiffness. Sodium Pentaborate pentahydrate powdered material which is produced in laboratory. Sodium Pentaborates anhydrite powdered material which is produced in laboratory.

2.2 Preparation

The SPB and SPA powdered materials were dried at 105 °C 1 hour with Memmert VO400 desiccator. Without drying the SPB, the air bubbles occur while injection processes in PP composites. The XPlore MC15 HT micro compounder set 175 °C, 200 rpm iPP and SPB mixed 10 minutes for each composite mixture. The XPlore IM12 injection machine set 180 °C and 10kN injection force with 170 °C mold temperature. The mold or the design of part is 4x100x100 mm test stick.

2.3 Characterization

The compounds are given at Table 1. Totally 11 sample had injected. Every sample prepared 10 pieces for tests [22]. The important point is that hard tests were tried at %5 and %10 compounds before and it is aimed at understanding the effects of high quantity of additives on polypropylene [5]. The other %0.5, %1, %1.5 quantity additives designed for understanding the trend curve of mechanical properties and microstructure changing way.

Table 1 Sodium pentaborate pentahydrate and anhydrate compounds details with Polypropylene

NAME	**MIX (%)**	**PP (gr)**	**SPP (gr)**	**SPA (gr)**
SPB05	0.5	8.955	0.045	-
SPB10	1	8.910	0.090	-
SPB15	1.5	8.865	0.135	-
SPB50	5	8.550	0.450	-
SPB100	10	8.100	0.900	-
SAN05	0.5	8.955	-	0.045
SAN10	1	8.910	-	0.090
SAN15	1.5	8.865	-	0.135
SAN50	5	8.550	-	0.450
SAN100	10	8.100	-	0.900
Reference PP	0	9.000	-	-

2.4 Mechanical Tests

The tensile experiment was performed with a tensile tester Instron 3300 stress testing device at a crosshead speed of 5 mm/min acc. to ASTM D 638 standard. Before tensile testing, the width and the thickness of the samples were measured with a caliper. The tensile modulus of the samples was determined at %0.5 strain. Five specimens of each sample were tested, and the mean values and standard deviations were calculated. All tests applied at 23±2 °C room temperature and the humidity were %55±5.

The Impact test was performed acc. to the ASTMD256 standard. The table feed rate and the cutter speed were 100 mm/min and 92 m/min. Before the impact testing, the depth and the width of the samples were measured with a digital caliper and micrometer. The samples were tested using DVT CD I impact tester. Ten Specimens of each sample were tested, and the mean values and standard deviations were calculated. All tests applied at 23±2 °C room temperature and the humidity were %55±5 [22].

2.5 FTIR Testing

The infrared spectra were recorded on a Perkin Elmer Spectrum 10.5.4 Version. The test only applied between PP, SAN15 and SPB15 comparison format. For each spectrum 64 consecutive scans with 4 cm^{-1} resolution were coded. The wavelength was around 450-4000 cm^{-1}. The background of spectrum was subtracted from the spectra obtained during the diffusion measurements. The spectrum Samples were measured in the form of 4±0,1mm thickness. Injection molded samples surfaces used for tests and applied at 23±2 °C room temperature and the humidity were %55±5 [23].

2.6 SEM testing

The SEM tests were applied Zeiss Gemini Sem 500 scanning electron microscope at 10kV. Nine samples (except SAN100 and SPB100) were tested. The samples were first cryogenically fractured in liquid nitrogen and then coated with a thin layer of gold/palladium at Leica EM ACE600. To observe the fracture behavior of samples, the tensile fracture/breakaway surfaces of the specimens were directly coated and examined by SEM. The tests applied at 23±2 °C room temperature and the humidity were %55±5 [24].

III. RESULTS AND DISCUSSIONS

3.1 Mechanical Tests

Polypropylene is a semi crystalline material and pure Borealis HE125MO homopolimer based isotactic polypropylene had used for comparison.

The results of the mechanical tensile tests of samples (for various filler concentrations) are given in Table 2. The standard deviation is maximum %5 each value at Table 2.

Table 2 Tensile test results of samples

Sample	**Modulus of elasticity E (MPa)**	**Yield strength σ_y (MPa)**	**Ultimate strength σ_u (MPa)**	**Yield strain ε (%)**
SPB05	1752.4±82	30.28±0.51	18.34±0.99	18.14±0.99
SPB10	1782.2±74	30.43±0.32	19.56±0.73	18.21±1.31
SPB15	1798.5±69	30.15±0.42	18.15±0.56	18.53±1.67
SPB50	1941.7±90	28.60±0.66	16.92±0.63	8.42±0.54
SPB100	1821.2±93	26.82±0.52	15.58±0.81	5.95±0.59
SAN05	1772.9±77	29.82±0.43	18.61±0.91	19.40±0.48
SAN10	1825.5±67	30.26±0.41	18.72±0.49	19.60±0.22
SAN50	2129.5±43	28.50±0.39	17.26±0.63	8.98±0.36
SAN100	2000.7±54	27.38±0.48	15.85±0.77	8.85±0.64
Reference PP	1732.0±62	29.19±0.70	19.74±0.81	18.84±1.44

The results of tensile stress graphs trend lines are also shown at Figure 1. The horizontal line gives the sample types, and the perpendicular line gives stress (MPa) at E, σ_y and σ_u graphs. At the ε graph, the horizontal line represents percentage elongation.

Elastic modules are always in a high trend with the addition of minerals which are sodium pentaborate pentahydrate and sodium pentaborate anhydrite. If talc additive mix with polypropylene, the elasticity modulus increases directly proportional with its quantity [25]. It also observed at calcite additives to polypropylene increase the elasticity modulus [26]. Generally mineral additives in polymeric materials increase the elasticity of polypropylene with direct proportion of quantities and particle sizes [27].

The tensile modulus increased to SPB05, SPB10, SAN05 and SAN10 samples then it started to decrease with %1.5 additives. %5 and %10 sodium pentaborate pentahydrate and anhydrate additive samples are decreasing dramatically because of proportion. The elasticity modules and the tensile strength critically depend on the stress transfer in the composites and the interfacial adhesion strength between polymer chains and particles. So that, if the rigidity changes with additive and has molecular relationship with polypropylene, the tensile strength is expected to increase. The elasticity modulus is up on the general stiffness and hardness of sample. The minerals rigidity is higher than polypropylene and the reason for that elasticity is expected to increase.

The ultimate yield strength decreases proportionally with the quantity of mineral additive. The samples are getting much rigid with increasing additive proportion, and it is expected to break away from lower stress. Parallel to this idea, the strain of yield decreases because the elasticity increases, and the chain bonds expected to breakaway easily. So, it is expected to decrease strain at higher proportion additive samples. When it is detailed the Figure 1, %0.5, %1 and %1.5 additives are near with pure iPP.

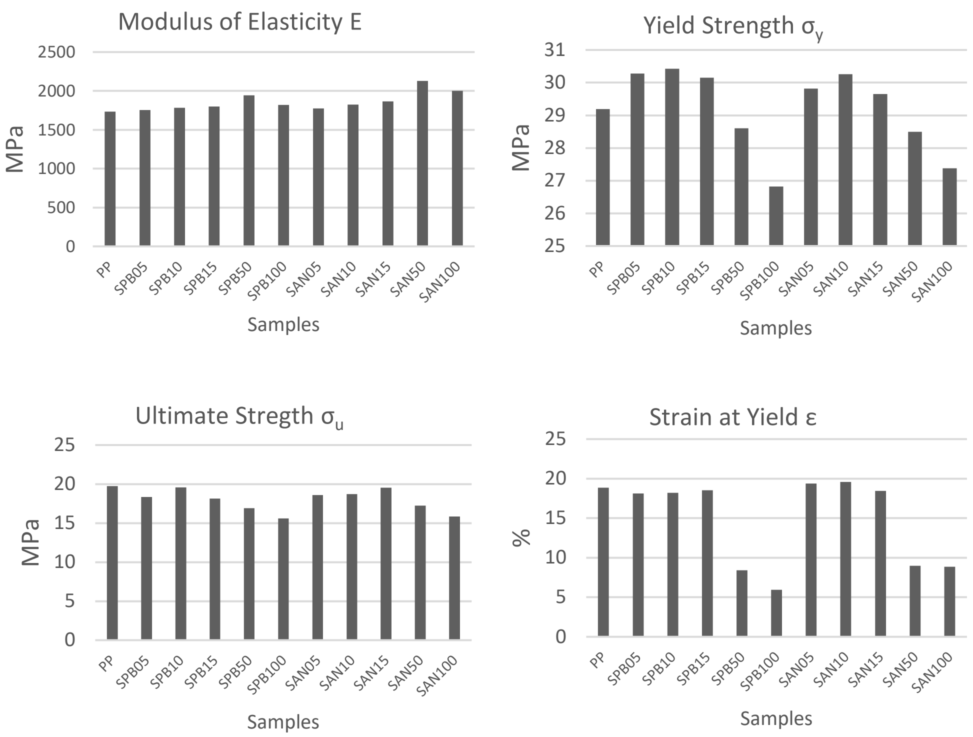

Figure 1. Graphs view of mechanical properties of samples

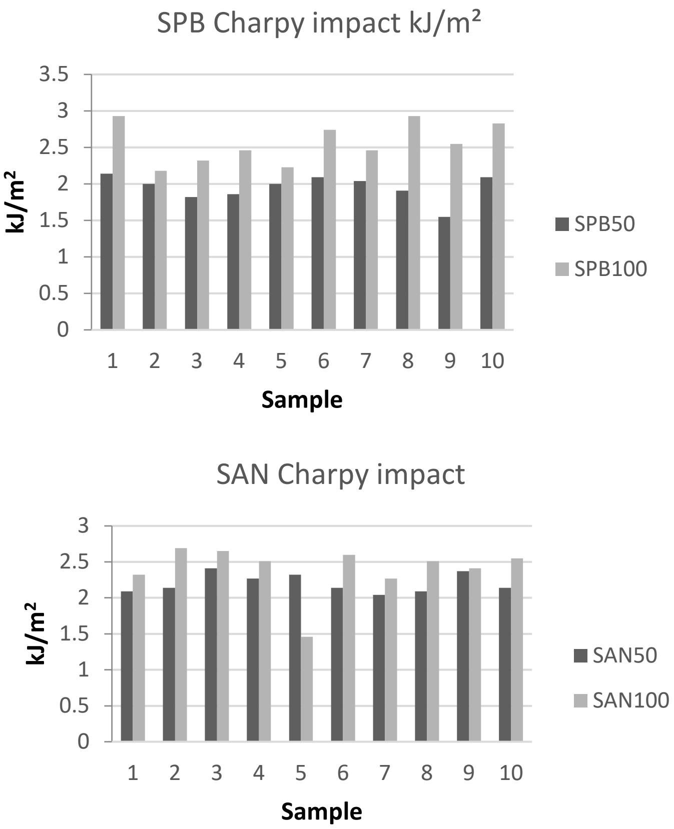

Figure 2. Charpy impact test results of SPB and SAN samples %5 and %10 additives.

The Charpy impact test to the samples are given at Figure 2. The horizontal line gives the sample numbers, and the perpendicular line gives absorbed energy at place kJ/m². The colors of the bars tell samples type. Except sample 5 at SAN Charpy impact graph, all values increase directly with the percentages of additives. It is expected to increase at surface tension because the density of material increases, and more rigid material adds in polypropylene [28]. There is no proportion between the additive percentages and Charpy impact values of samples because specimens cannot be produced same homogeneity.

3.2 FTIR

IR spectroscopy has been widely used as an analytical and characterization tool in the polymer field [18]. It is well known that there is a great complexity in the spectra obtained from polymers due to the existence of overlapped bands, changes in their shapes, and displacements all along the frequency axis. These facts may be a serious problem when trying to quantify accurately.

Upon processing the semi crystalline nature of the polymer (iPP) could have changed in the crystalline content. Then an overlapping phenomenon is expected to occur between the vibration modes of groups present in the amorphous regions and those belonging to crystalline regions. Thus, it would be very important to choose adequate reference bands to avoid skewness in handles data, which would lead to wrong results [16]. For these reasons the experiments made between pure iPP and SAN15 and SPB15 with comparison method at Figure 3.

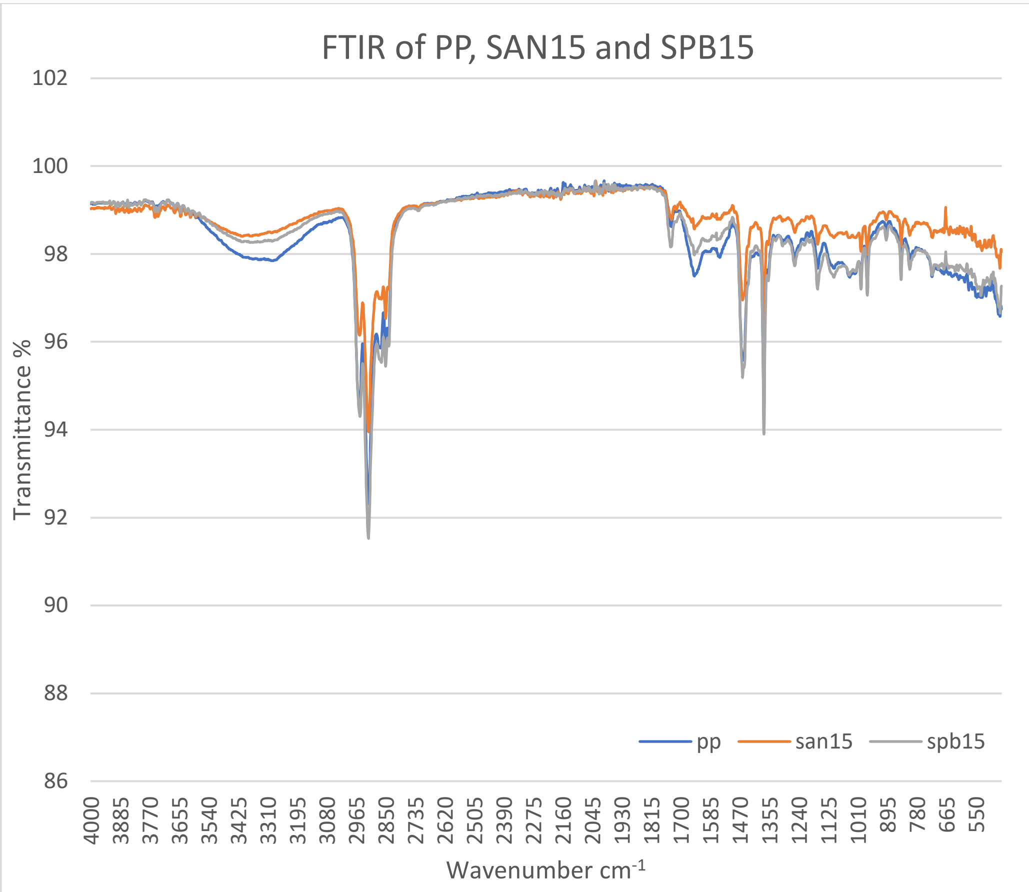

Figure 3. FTIR test of PP, SAN15 and SPB15

Considering the general results, in terms of their differences between PP and SAN15 is 97.1434%. In other words, approximately 3% difference was obtained between San and PP contribution. There is 98,4466% similarity between SPB15 and PP. According to these results, Sodium pentaborate differentiates PP material in the internal structure. The molecular structures are shared at Figure 4.

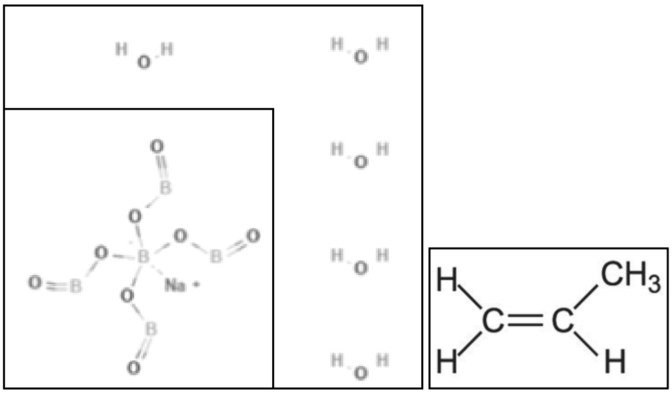

Figure 4. Sodium Pentaborate and Polypropylene monomer molecular structures

At Figure 3, differentiation can be seen as an O-H band between 3600-3000 cm^{-1}. SPB15 is lower than PP, SAN 15 is lower than both. In that region where O-H bonds are represented, this decrease can be interpreted as the effect of sodium pentaborate. This condition means sodium pentaborate molecules break the hydrogen bonds from polypropylene chains because of oxygen atoms in structure [23]. SAN15 has lower value than SPB15 because of the water molecules. The SPB15 got water molecules, and they prevent the O-H bonds directly, but it could be cause H dipol bonds. It can be seen at Figure 5 [28].

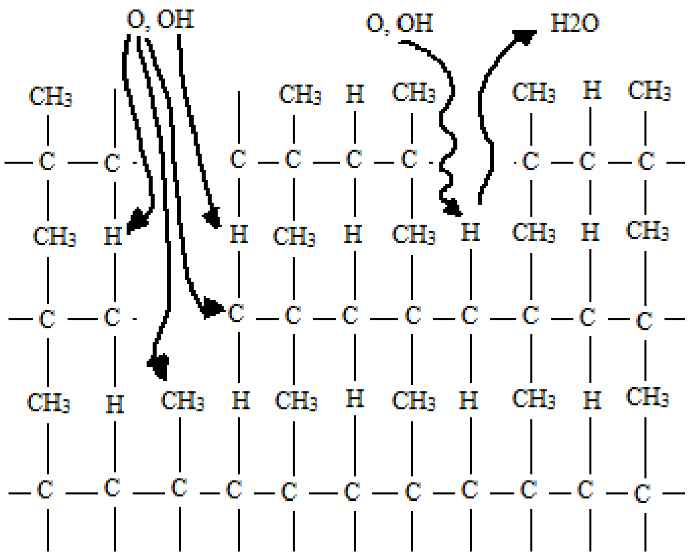

Figure 5. O and OH atoms' molecular effects on polypropylene chains. [28]

CH_3 and CH_2 sharp peaks are observed in the region of 2960-2830 cm^{-1}. The region is important because the perpendicular strong bonds of CH_2 and CH_3 increases the mechanical strength with ramification effect [29]. The

SAN15 is at lower transmittance value at that region too. The anhydrite form could decrease the ramification with blocking CH_3 bonds.

At the 1750-1450 cm^{-1} region, both SAN15 and SPB15 decreased the C=C bonds. This effect is greater than SAN15 because boric acid could interact with C bonds. This means oxidation at PP [30].

Below 1400 cm^{-1} region is fingerprint area. The change in this region indicates that the material (PP) has changed. There are 1459, 1376, 1255, 1168, 999, 974, 842, 806 and 720 cm^{-1} peaks in this region [31]. In all these peaks, while SPB15 has improved, it is observed that SAN15 is at worse levels.

Sodium pentaborate pentahydrate and anhydrite 1,5% additive forms of PP had not had sharp absorption peaks but have changes at its FTIR tests.

3.3 SEM

Figure 6 shows the morphology of the fractured areas of samples. In general, it is seen that the sodium pentaborate pentahydrate and anhydrite particles spread inside the polypropylene and settled between the PP fibers structures with white color.

At Figure 6, the “a” and “b” pictures shows a pure polypropylene. There is no white dots. The fractions way is crosswise. The sodium pentaborate is shown at “c” and “d” pictures. Approximately 15x6 μm particle zoomed x50 times and Figure 6-d shows the agglomerated structure of it. It is also seen whiter than PP structure. The fracture surfaces of pure PP layers are much smoother than other samples.

The polypropylene fibers and sodium pentaborate pentahydrate particles are discernable at Figure 6-e with 2μm zoomed image. Additive particles come between the PP fibers, and it cause fibers/chains of polypropylene to tighten and interlaced each other. That means nucleating effect of the SPB nanoparticles effects the structure that the size of crystals. The pentaborate nanoparticle has change the chain/fiber way at “e”. So that is one of the reasons for rising tensile strength [32]. The bigger particle of SPB is visualized at Figure 6-f.

The fraction ways of SPB15 at Figure 6-g are more horizontal than PP. This means the additives make part much solid that the cross dilacerations are decreased. Figure 6-h shows the fiber structure, and it became thicker or thinner phase randomly depends on the position of particles with additive percentage.

At Figure 6, “i” and “j” tells that if the percentage increase, the fiber structure covered with agglomerated sodium pentaborate particles. This also explains the surface tension increase. Besides, the density rise with additive causes the elasticity grows.

The anhydrite form is shown at Figure 6-l. The particle is positioned between the PP fibers. Without water, the pentaborate particles scattered a little bit more in comparison with pentahydrate form. At Figure 6-m, fractures are more horizontal. Figure 6-n shows us the chain bonds with pentaborate at 400 nm. The chains/fibers bonded with sodium pentaborate. The FTIR test is verified with SEM with this image.

At Figure 6 “o” and “p”, the 5% percent of anhydrite form visualized. The point is flocculation of SPA is seen at “p”. This causes irregular mechanical strength and decreasing tensile strength [26]. It is one of the reasons of -

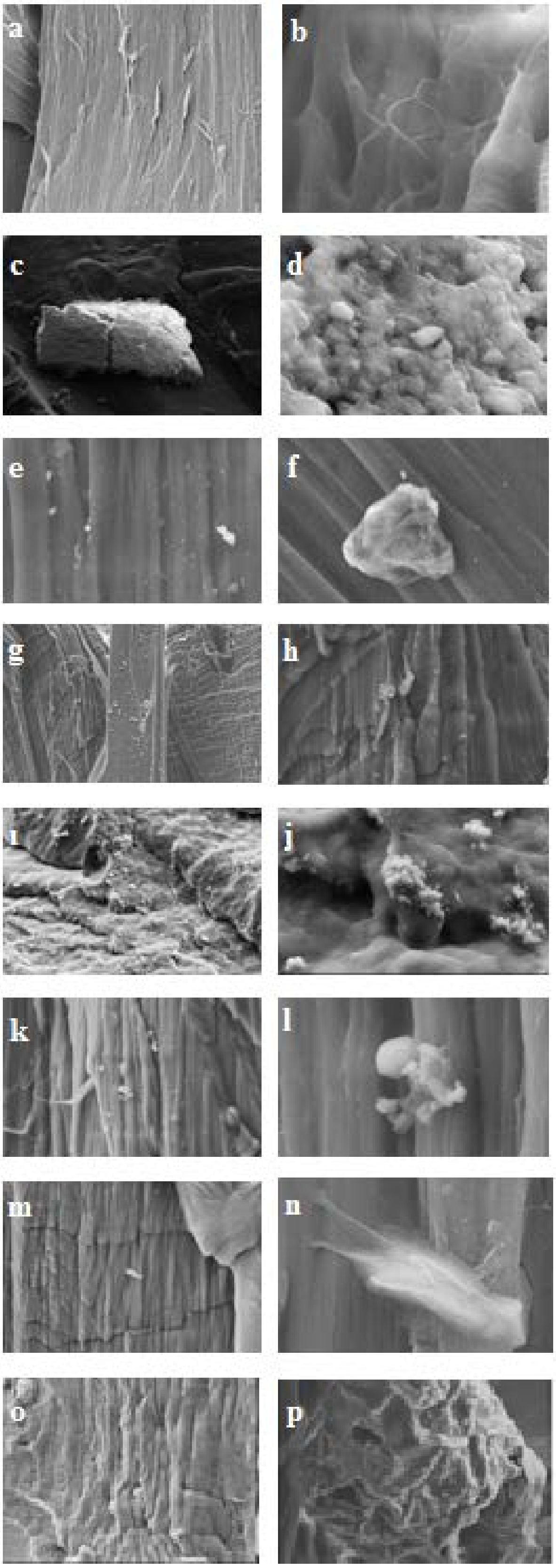

Figure 6. a) x500 PP 40µm, b) x30000 PP 1µm, c) x1000 Sodium pentaborate additive 20µm, d) x50000 Sodium pentaborate additive 400nm, e) x10000 SPB10 2µm, f) x5000 SPB10 4µm, g) x1000 SPB15 20µm, h)x5000 SPB15 4µm, i) x2500 SPB50 10µm j) x10000 SPB50 4µm, k) x5000 SAN10 4µm l) x30000 SAN10 1µm m) x5000 SAN15 4µm n) x50460 SAN15 400nm, o) x2500 SAN50 10µm p) x5000 SAN50 4µm SEM images of fracture surfaces of samples

decreasing strain at yield values. The structure is less agglomerated than SPB50 because of the lack of water molecules.

Sodium pentaborate ($NaB_5O_8 \cdot 5H_2O$) nanoparticles exhibit a significant tendency for agglomeration during dispersion due to their high surface energies. This phenomenon can adversely affect the material's homogeneity and final mechanical performance, particularly in surface coating processes such as metal matrix composites or micro-arc oxidation (MAO) [33].

Agglomeration leads to the formation of porous, brittle regions with low bonding strength, as it increases the contact points per unit surface area of the particles. This can result in irregular distribution of properties such as hardness and wear resistance, as well as the emergence of localized weak points. In the absence of appropriate dispersion techniques (such as ultrasonic mixing, pH control, or surface modification), negative effects may arise when the optimum additive level is exceeded due to the solubility and clustering tendency of salts like sodium pentaborate, which possess hydrophilic characteristics. However, it has also been reported that limited agglomeration can create micro-barriers that restrict crack propagation in certain cases, thereby demonstrating a positive effect at controlled additive levels [34]. Therefore, in the design of sodium pentaborate-containing systems, particle size, loading ratio, and distribution method should be optimized in conjunction.

All in all, the microstructure of polypropylene is changed proportionally by percentage of sodium pentaborate additives as seen at Figure 6.

IV. CONCLUSIONS

The mechanical effects of sodium pentaborate pentahydrate and anhydrite additive in polypropylene has been studied. With changing degrees of filler content, the alteration of mechanical strength of Polypropylene – sodium pentaborate composite matrix was found. The observed increase in the tensile strength ranged 4,2% with %1 addition of sodium pentaborate pentahydrate. The modulus of elasticity increased with the rising of additive proportion. The relationship between additive proportion and mechanical change is scrutinized as yield at strain and ultimate strength values too. Simultaneously, an increased brittleness was obtained which was verified with the charpy effect tests.

These changing mechanical properties effects were ascribed to the influence of the dispersion and micro particles in the polypropylene matrix. The mutual synergistic effect of admixtures, which resulted in the differentiation of bonds between individual particles and polymer matrix with FTIR tests. The sodium pentaborate penetration to the fiber/chain structure of polypropylene was examined and associated with mechanical impacts. In microstructure, the dissemination and localization of sodium pentaborate particles to polypropylene were evinced through the instrument of SEM. It is observed that the agglomeration internal structure occurs if the proportion of sodium pentaborate increases. Respectively, sodium pentaborate pentahydrate increases the mechanical properties better than anhydrite form.

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