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EFFECT OF SHELLAC ON STRENGTH AND WATER RESISTANCE PROPERTIES OF Na_2SiO_3 AS WOOD ADHESIVE

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ABSTRACT

In different types of industries, a lot of adhesives are used, which are harmful to living beings and are the main source of environmental pollution. Among inorganic adhesives, sodium silicate is very popular due to its excellent binding qualities. It is odorless, fireproof, non-toxic, and free from volatile organic compounds. It is affordable and simple to use, but its low water resistance limits its large-scale commercial utilization in the wood-based sector. To strengthen the link between teak wood and sodium silicate, three different sets of Na_2SiO_3 adhesive—pure Na_2SiO_3, modified Na_2SiO_3 by using silica gel G (as curing agent), and modified Na_2SiO_3, having different concentrations of shellac (2 -10%) were prepared in the present study. According to the findings, the best combination for tensile strength and moisture resistance is modified sodium silicate with 6% and 8% shellac, respectively. The FT-IR data demonstrated that shellac had effectively intermixed within the structure of the silicate and had good thermal stability in the 27^0–802^0C temperature range, which is also confirmed by the results of XRD, TGA, and DTG.

Keywords: Adhesive, Na_2SiO_3, Shellac, Water Resistance, Tensile Strength, Silica Gel G.

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INTRODUCTION

Inorganic adhesives have gained more and more attention in recent years due to their low-cost, non-burning, eco-friendly nature than organic adhesives.[1,2] The most promising inorganic adhesive in the market is silicate adhesive or sodium silicate. Sodium silicate, commonly referred to as water glass, has a lot of excellent properties. It shows very good adhesion, can be prepared from natural resources easily, and is very easy to handle.[3,4] It is fireproof, non-explosive, and free from toxic substances.[5-7] Sodium silicate is free from volatile organic compounds, has low adhesive stress, is difficult to burn, and exhibits strong temperature resistance. It can be used for bonding in the metals, ceramics, glass, stone, and wood industries.[8,9] Due to the hygroscopic nature of sodium silicate, its large-scale commercial application as a wood adhesive is limited.[10,11] Also, its glue line is brittle and inelastic. The uses of Na_2SiO_3 as a gluing material have received few investigations.[12-15] The change in adhesive strength of sodium silicate was observed by incorporating silicate fumes, diamond powder, phenol formaldehyde resin, guar gum, nanoparticles, carboxymethylcellulose, gum arabic, ammonium stearate, cellulose nanofibrils, urea-glucose-melamine resin, CO_2, etc.[16-26] After reviewing the results of previous literature, it might be possible to achieve high-performance silicate adhesive (good water resistance and better bonding strength) through modification. In the present research, the improvement of the bonding properties and moisture resistance of sodium silicate as a wood adhesive is studied by using silica gel G as a curing agent and shellac as an additive. The water resistance and bonding strength of the Na_2SiO_3 adhesive were evaluated in order to assess its adhesion properties. Exploring the interaction of silica gel G, shellac, and Na_2SiO_3 in the adhesive structure, it was discovered that the addition of shellac to Na_2SiO_3 adhesive improved its quality, as evidenced by the cured morphology and thermal characteristics.

EXPERIMENTAL

Materials

Sodium Silicate (SS)

A Sodium silicate solution of grade 52° Be suitable for adhesive properties. It was purchased from O P Industry in the Jaipur district of Rajasthan (India). The chemical analysis of sodium silicate is: Na_2O =13.65%, SiO_2 = 33.66%, Viscosity = 1553.23, mPa.s, ratio of SiO_2/Na_2O weight = 2.47:1, specific gravity =1.54, pH = 11.70, total solids = 26%.

http://doi.org/10.31788/RJC.2024.1748654

Shellac
Shellac of the purest grade was procured from a local bangle shop in Jaipur.

Silica gel G (curing agent)
The store of the Chemistry Department, Rajasthan University, Jaipur, provided Silica gel G, which contained 13% $CaSO_4$.

Wood
Teak wood *(Tectona grandis)* was purchased from the Balaji Timber Industry, Jaipur.

Methods
Preparation of Samples of Wood
Wood samples of 150x25x3 mm containing 13% moisture were prepared.[27,28]

Preparation of MSS (Modified Na_2SiO_3 Adhesive)
First of all, 80 ml of Na_2SiO_3 was poured into a beaker, then 20 g of curing agent silica gel G was mixed with it, and then this was continuously stirred to obtain a homogeneous adhesive.

Preparation of Shellac Solution
A saturated solution of finely powdered shellac was prepared by using denatured alcohol. For this, in a clean and dry beaker, powdered shellac was mixed with 100 mL of denatured alcohol to form a saturated solution, and then the shellac suspension was boiled in a water bath at 60ºC for 30 minutes under continuous stirring to form a homogeneous solution.

Preparation of Sodium Silicate Adhesive without and with Shellac (MSSS)
Table-1 represents the different samples of the experiment. Figure-1 shows the prepared modified Na_2SiO_3 adhesives with different percentages of shellac.

Table-1: The Samples of Experiments

Component	SS (Simple sodium silicate)	MSS	MSSS (containing different % of Shellac)				
			2%	4%	6%	8%	10%
Sodium silicate/mL	100	80	80	80	80	80	80
Silica gel G/g	0	20	20	20	20	20	20
Shellac/mL	0	0	2	4	6	8	10

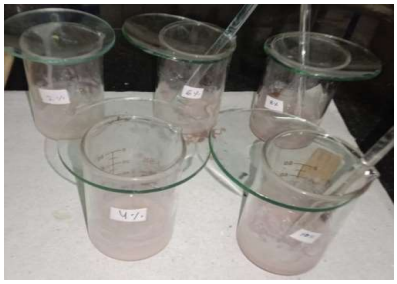


Fig.-1: Different Types of MSSS (2% to 10%)

Moisture Resistance Investigation
Teak wood blocks were glued by a double coating of SS, MSS, and MSSS containing different percentages of shellac and pressed under a 0.5-0.9 MPa load of static pressure at thirty degrees centigrade for twenty-four hours (Fig.-2). The difference in weight before and after 24-hour H_2O immersion was recorded, and the ratio of this was used to compute the water absorption rate at various time intervals.[29,30] Results are reported in Fig.-4.

Tensile Strength Investigation
To find out the tensile strength, Teak wood blocks were double-glued using SS, MSS, and MSSS with shellac ranging from 2% to 10%. These wood blocks were then subjected to a static pressure load of 0.5–

0.9 MPa at 30°C for 24 hours, and then these were examined by using a tensile strength testing machine (UTM) (Fig.-3). Findings are reported in Fig.-5.

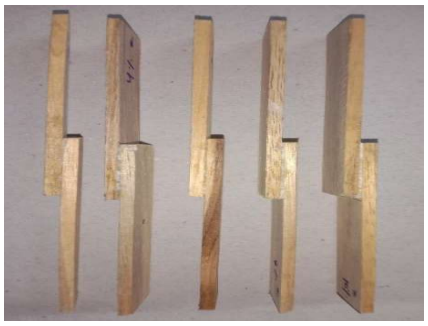


Fig.-2: Prepared Wood Block Samples for Moisture Resistance Testing

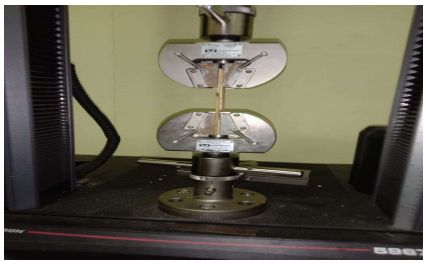


Fig.-3: Determination of Tensile Strength of Na_2SiO_3 Adhesive on Wood Blocks

Every experimental technique was carried out six times, after that the average outcomes were presented.

Spectroscopic Investigations

Fourier Transform-Infrared Spectroscopy (FT-IR): For bonding and characterization of the adhesive, FT-IR spectroscopy is the most useful technique. To obtain the FT-IR spectrum, each sample was scanned 32 times using the ATR mode.

X-ray Diffraction (XRD): Powder X-ray diffraction (PXRD) measurements were used to determine the crystal structures of the samples by using Cu Kα monochromatized radiation at 40 mA and 40 kV. The X-ray profiles were collected between 10^0 and 90^0 with a count time of 1 s/step.

Thermogravimetric (TGA-DTG): To find out the thermal stability of the adhesive samples (TGA and DTG), thermal heat was provided to samples from 27^0C to 800 °C at a rate of 15 °C per minute using N_2 gas flowing at a rate of 20 mL per minute.

RESULTS AND DISCUSSION

Concept of Sodium Silicate Modification

Effect of Curing Agent Silica Gel G on the Adhesion Qualities of Na_2SiO_3 Wood Adhesive

After fully curing, a single component of Na_2SiO_3 adhesive turns into a translucent, brittle substance. According to preliminary studies, when it is employed as a wood adhesive, curing defects, including bubbles and stress cracks, are easily identified in the binding line. To make Na_2SiO_3 wood adhesive suitable for wood adhesion, it is therefore important to lessen its brittleness by adding the appropriate filler, curing agent, or skeleton material. A curing agent is a crucial part of adhesive, which can immediately interact with the binding material, resulting in the production of a tough and strong 3-D network structure, changing the properties of the adhesive. The mechanical and binding strength of the wood adhesive is also affected by the quantity of curing adhesive. The curing agent hydrolyzes into silicic acid in an aqueous solution of sodium silicate, after that, it readily dries and solidifies. Silica gel G is used as a film-forming and curing agent for Na_2SiO_3 wood adhesive in the present work. It increases the formation of silicic acid in SS. The chemical reactions related to curing are as follows:

$$Na_2O.nSiO_2 + (2n+1)\ H_2O \longrightarrow 2NaOH + nSi(OH)_4$$

$$Na_2O.nSiO_2 + 2n\ H_2O + CO_2 \longrightarrow Na_2CO_3 + nSi(OH)_4$$
$$2SiO_2 + 4H_2O \longrightarrow 2Si(OH)_4$$
Silica gel — Silicic acid

$Si(OH)_4$, the aforementioned product, can auto-agglutinate to generate multi-polysilicic acid. The inorganic film is formed by dehydrating and condensing the -Si-O-Si-chain network structure that is produced by multi-polysilicon acid. In addition, silica gel G contains a trace quantity of calcium sulfate, which quickly hydrates to become gypsum, a very effective binder, further accelerating the curing process and boosting the product's strength.[31]

$$CaSO_4 + 2H_2O \longrightarrow CaSO_4.2H_2O$$
Gypsum

Effect of Shellac

Shellac is a natural resin with a complex mixture of ester, polyester, and polyhydroxy acids. Hard resin, soft resin, and wax are the main three constituents of shellac.[32] The resinous constituents of shellac contain hydroxy acids, and their polar groups are present at the interface of the molecule.[33] It is a mixture of several polar and non-polar components in a molecule. When shellac solution is mixed with sodium silicate solution, it yields a complex mixture of polymerization between silicic acid (Si-OH) and the aliphatic and alicyclic hydroxy acids (–OH) of shellac, and enhancing the formation of –Si-O-Si- network in the adhesive and will promote curing and improving water resistance. Due to excellent abrasion resistance, insoluble in water, good adhesion, oil resistance, durability, and dielectric behavior, Shellac was selected as an additive to modify the adhesive performance of sodium silicate by using its excellent properties and waterproof character.

Water Resistance Property

The water resistance property is inversely proportional to the water absorption property. The water absorption rates of SS, MSS, and MSSS with varying amounts of shellac solution were studied at different periods (three, six, nine, twelve, fifteen, eighteen, twenty-one, and twenty-four hours). The moisture absorption rate of SS after 3 hours was 2.68%, and then it began to dissolve slowly in the water, hence its water absorption rate decreased to 2.60%. As time passed, it completely dissolved into water due to its water-soluble nature; therefore, between 6-9 hours of water immersion, teak wood blocks glued by simple sodium silicate were completely separated. While wood blocks joined by MSS and MSSS containing different amounts of shellac were not separated even after 24 hours of water immersion. It was clear that as time passed, the water absorption rate also increased for each type of adhesive. Between different percentages of shellac additive, it was found that modified sodium silicate with 8% shellac exhibited a lower water absorption rate than other compositions (Fig.-4).

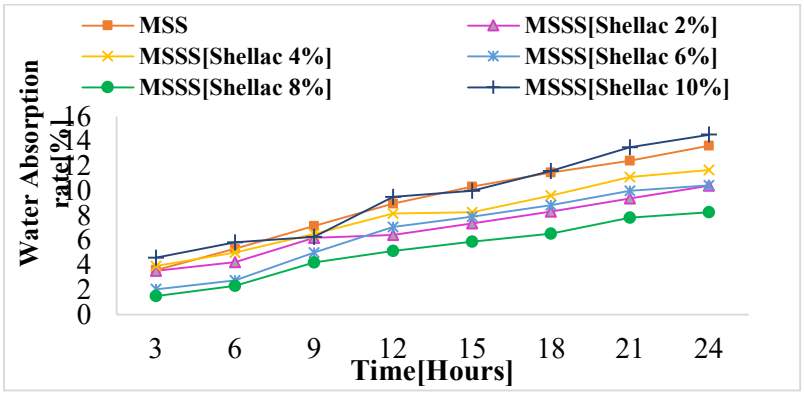


Fig.4: Water Absorption Rate of MSS and MSSS having Different % of Shellac

Tensile Strength (or Bonding Strength)

Figure-5 represents the adhesive performance of SS, MSS, and MSSS having different % of shellac (2% to 10%). The tensile strength is 3.87 MPa for SS, which is the minimum for others. The value of tensile

strength is enhanced to 7.90 for MSS. For MSSS, the value of tensile strength first increases from 2% to 6% and then decreases from 6% to 10%. In comparison to all Na_2SiO_3 wood adhesives, the highest tensile strength of 9.05 MPa was found for 6% MSSS. Therefore, the bonding strength of the sodium silicate was enhanced to 9.05 MPa from 3.87 MPa.

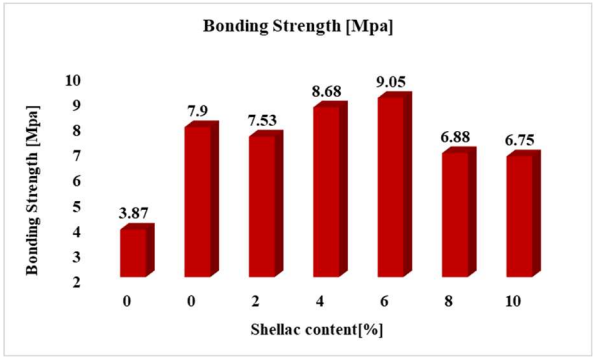


Fig.-5: Tensile Strength of Sodium Silicate, MSS and MSSS (2 % - 10%)

The curves of experimental failure loads with extensions of sodium silicate, Modified Sodium Silicate, and MSSS of 2-10% shellac content, represented in Fig.-6. Fig.-6 (a) showed that at maximum tensile strength highest failure load was found. Fig.-6 (b) represented that the load at maximum tensile strength was found maximum 1488.29 N for MSSS [6%]. Maximum tensile stress is shown at 6% MSSS, represented in Fig.-7 (a) and in Fig.-7 (b) elongation % is decreases from MSS to MSSS [2% to 10%].

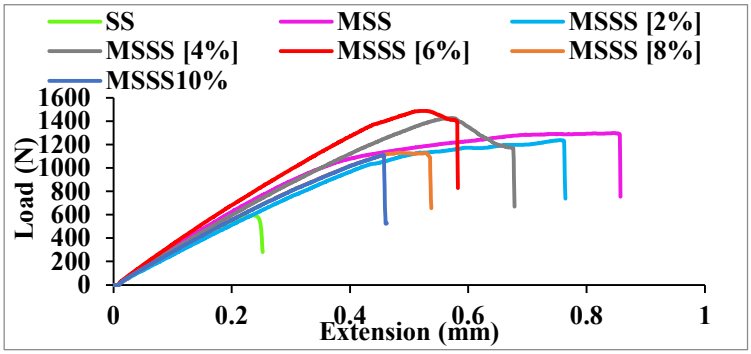


Fig.-6 (a): Load (N) Versus Extension (mm) for Wood Blocks Jointed by using Sodium Silicate, MSS, and MSSS (containing 2% to 10% shellac)

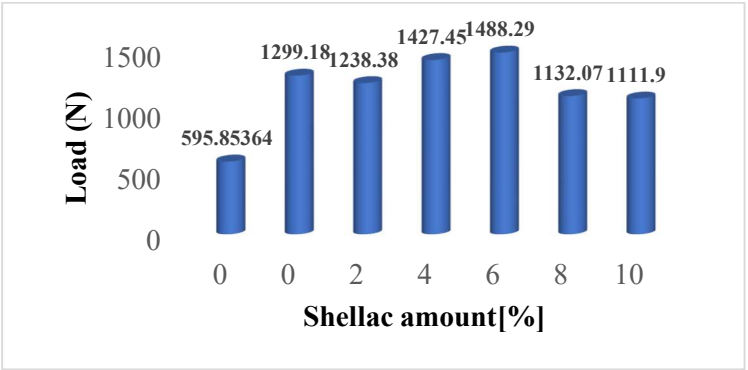
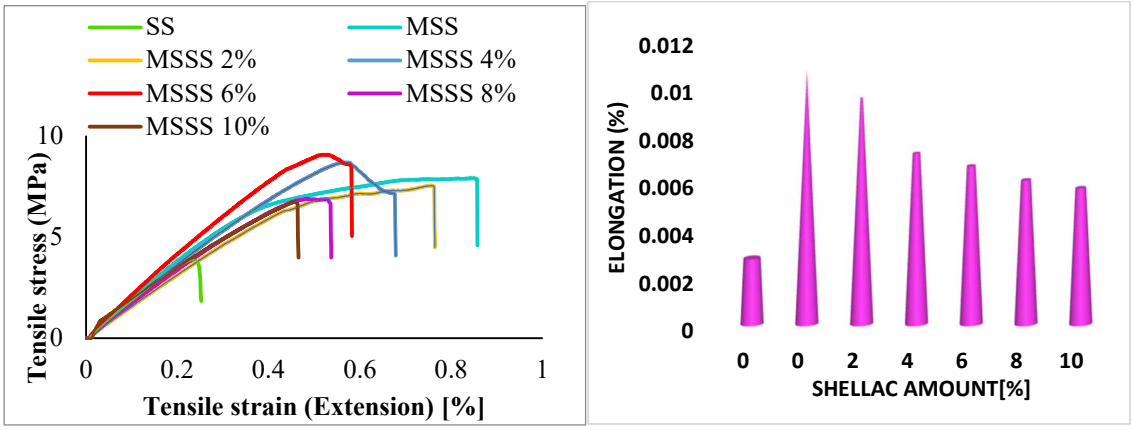


Fig.-6 (b): Shellac amount [%] Verses Load (N) [at maximum tensile strength] for SS, MSS, and MSSS (2% to 10%)

24-hour H_2O Absorption Rate vs Strength of Bonding

The twenty-four-hour H_2O absorption rate and bonding strength of Modified Na_2SiO_3 (13.64%, 7.90 MPa) are better than SS (separated, 3.87 MPa). As the amount of shellac in the modified Na_2SiO_3 solution

increased, the tensile strength and the twenty-four-hour rate of H_2O absorption both first increased and then reduced. When the shellac is 6% in MSSS, the bonding strength is maximum (9.05 MPa), while the water absorption rate for twenty-four hours was 10.43%.



(a) (b)

Fig.-7 (a): Tensile Strain Versus Tensile Stress for Wood Joint Specimen by using Simple Sodium Silicate, Modified Sodium Silicate, and Various Types of MSSS Containing 2 % to 10% Shellac

Fig.-7 (b): Shellac content [%] Versus Elongation [%] (at maximum load) for SS, MSS, MSSS [2% to10%]

A tensile strength of 6.88 MPa and the lowest 24-hour H_2O absorption rate of 8.26% were achieved at 8% shellac dosage. The bonding strength decreased as the shellac content continued to increase, and the 24-hour water absorption rate also started to rise, suggesting that using too much shellac was detrimental to adhesion. At high concentrations of shellac with sodium silicate solution, the acid value decreased and the ester value increased, indicating the self-esterification of shellac increased with increasing the concentration of shellac in solution. Since hydrolyzed shellac has a greater number of polar groups (-OH, -COOH) at high concentrations, this results in intramolecular interactions through hydrogen bonding via ester formation. Therefore, it concluded that as the amount of shellac increased, cross-linking within shellac molecules occurred, therefore, less amount of shellac was available to react with sodium silicate to form an –O-Si-O- bond. The results showed that the incorporation of silica gel G as a curing substance and shellac as an additive to sodium silicate greatly increased its 24-hour water absorption rate and bonding strength. Results are reported in Fig.-8.

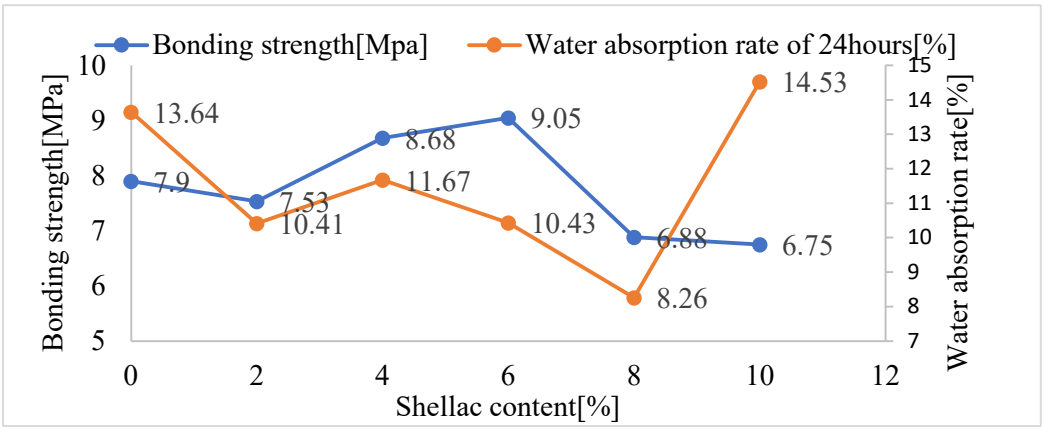


Fig.-8: Twenty-Four-Hour Water Absorption Rate and Bonding Strength of MSS and MSSS (Having Shellac 2% to 10%)

FT-IR Analysis

The compiled FT-IR pattern of SS, MSS, and 6% MSSS samples is shown in Fig.-9. A broad stretching absorption peak at 3257.25cm^{-1} was of the Si-OH stretching vibration band in SS. After modification, this

peak was weakened due to the consumption of Si-OH in Si-O-Si bond formation. It is suggested that the incorporation of curing agent and shellac promote Si-O-Si bond formation. A small peak related to the $-CH_2-$ stretching vibration band is clearly visible in MSSS at 2970.35 cm^{-1}. The CO_2 absorption peaks were clearly observed in SS and in MSSS at 2326.15 $cm^{-1}$and 2323.62 cm^{-1} respectively. This was also noticed that the peaks at 1645.01 $cm^{-1}$and 981.22 cm^{-1} were Si-O bond stretching and bending vibration, respectively. It is also noticed that Si-O-Si bending vibration peak area is a little more in MSSS adhesive compared to remaining both spectra, indicating that different functional groups of shellac interact with Si-OH and form Si-O-Si bonds networks more, which enhances the bonding capacity of the silicate.
In SS and MSSS, a strong characteristic CO_3^{2-} peak appeared at 1440.79 cm^{-1} and 1440.28 cm^{-1} respectively. This implied that the subsequent chemical reaction had taken place during the curing process, resulting in the surface layer of cured adhesive.

$$Na_2O.mSiO_2.\ nH_2O + CO_2 \longrightarrow Na_2CO_3 + mSi(OH)_4.nH_2O$$

An amount of the free -OH groups bound with sodium silicate reacted with shellac and produced Si-O-C bonds, as evidenced by a medium-sized but sharp peak that occurred at 1216.96 cm^{-1} in the MSSS. Vibration peaks of Si-O-Si appeared at 981.22 cm^{-1} in SS. In MSSS a strong vibration peak of Si-O was strongly noticeable at 982.44 cm^{-1}, and at 427.47 cm^{-1} indicating that, compared to SS and MSS, combined chemicals in MSSS can further enhance the adhesive curing process to increase its bonding strength. The presence of these peaks showed that shellac with curing agent had a chemical reaction with Na_2SiO_3 adhesive and the shellac was effectively intermixed into silicate.

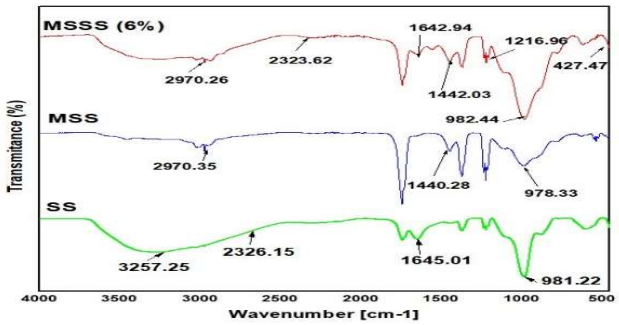


Fig.-9: FT-IR Spectra of SS, MSS and 6% MSSS

XRD

The XRD spectrum showed (Fig.-10) that there is a change in crystallinity in MSS and MSSS due to modification compared to pure SS. The modification of Sodium silicate adhesive with amorphous silica gel G and shellac degraded its crystallinity from SS to MSS and MSSS, respectively. The XRD spectrum indicated that the SS degraded in amorphous, after the modification with silica gel G and shellac from its pure crystalline structure, showing some crystalline peaks in SS 2θ around 30.15◦, 34◦, 35.25◦, 38◦, 40◦, 41◦, and many more, while a broad band 2θ around 27◦ in MSS and 27.17◦ in 6% MSSS.

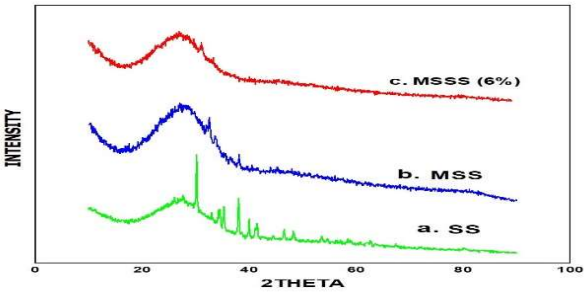


Fig.-10: XRD Spectra of SS, MSS, and 6% MSSS

TGA Analysis

The TGA analysis results confirmed the enhanced molecular structure of MSS and 6% MSSS wood adhesive displayed in Fig.-11. The total weight loss of MSS and 6% MSSS is 30.74% and 30.09%, respectively, while the total weight loss is 77.99% for simple sodium silicate wood adhesive.

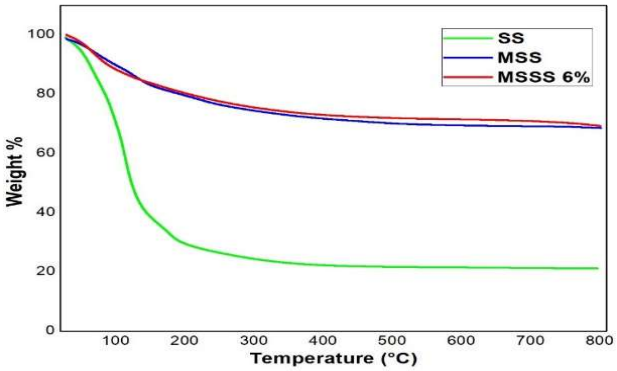


Fig.-11: TGA Curves of SS, MSS and 6% MSSS

It explains that the temperature stability of modified sodium silicate is 0.65% less than modified sodium silicate with shellac wood adhesive. It is also clear that modified sodium silicate wood adhesive without and with shellac has better thermal stability than simple sodium silicate in the studied temperature range (27 ^{0}C ~ 802^{0}C). The adhesive stability against high heat validates its high strength of bonding and moisture resistance, suggesting that Na_2SiO_3, silica gel G, and shellac-prepared networks of cross-linked structure in excess amounts, forming the new modified Na_2SiO_3 more stable towards thermal variations and can be used in a wider temperature range.

DTG Analysis

DTG analysis results of simple sodium silicate and after modification were presented in Fig.-12. It is easily noticed that simple Na_2SiO_3 reached the maximum rate of thermal decomposition around 116.36^{0}C, while for MSS and 6% MSSS adhesive, it is 75.35^{0}C, and 125.38 ^{0}C, respectively. For MSS and MSSS, DTG peak height and peak areas are both much smaller than those of simple sodium silicate. Therefore, it can be concluded that MSSS is more thermally stable than MSS. This suggests that the adhesive system exhibited greater thermal stability and a decreased rate of thermal breakdown after compound modification.

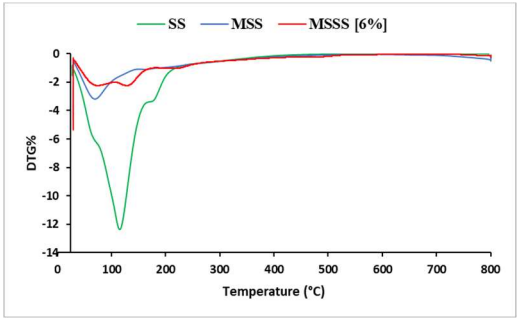


Fig.-12: Derivative Thermogram of SS, MSS and 6% MSSS

CONCLUSION

In comparison to simple sodium silicate adhesive, MSS and MSSS demonstrated better bonding strength and increased moisture resistance, hence goal of the modification was accomplished. The enhanced working capacity of modified Na_2SiO_3 as a binder is due to its improved molecular structure and thermal stability. Compared to MSS, the twenty-four-hour H_2O absorption rate fails in simple Na_2SiO_3 within 6-9 hours, and wood blocks jointed by SS were separated. In comparison to Na_2SiO_3, the twenty-four-hour water absorption rate of modified sodium silicate is 13.64% and wood blocks were still attached, and MSSS having 8% shellac was reported to have the lowest 24-hour water absorption rate, which improved water resistance by 36.95% from MSS. MSS bonding strength was enhanced by 104.13% than SS, and 6% MSSS bonding strength was improved by 14.55% more than MSS. In comparison to modified sodium silicate, the strength of bonding and moisture resistance properties of MSSS were enhanced by 14.55% and 36.95%, respectively. The overall outcomes showed that water resistance as well as bonding strength of Na_2SiO_3

have been significantly improved by incorporating shellac in MSS adhesive containing silica gel G. It is also recommended that to obtain improved adhesive qualities, the optimized Na_2SiO_3 formula with shellac should be 20 g of silica gel, 80 g of water glass, and 6-8% shellac of the weight of modified Na_2SiO_3 solution.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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