

Effect of Microwave Heating on Increasing Strength of Soda-Lime Glass by KNO_3 -Calcined Kaolin Slurry Spray Coating Technique

Pathomporn Ussawanawachat¹, Nitikan Kititarakul¹ and Apirat Theerapapvisetpong¹ *

¹*Upcycled Materials from Industrial and Agricultural Wastes Research Unit, Department of Materials Science, Faculty of Science, Chulalongkorn University, Bangkok, Thailand*

*Corresponding author e-mail : apirat.t@chula.ac.th

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Abstract

This research investigated the effect of microwave heating on increasing the strength of soda-lime glass using a KNO_3 -calcined kaolin slurry spray coating technique. Glass samples were coated using a spray technique and subjected to 1200W microwave heating at 40-50% power for 10-20 minutes in an alumina chamber with silicon carbide susceptor, followed by electric furnace treatment at 470°C for 0-4 hours. The mechanical properties were evaluated through flexural strength testing and Vickers microhardness measurements. Light transmission was analyzed using UV-Vis spectrophotometry, and potassium ion diffusion was examined using SEM-EDS. Results showed that microwave heating at 40% power for 10 minutes followed by electric furnace treatment at 470°C for 4 hours yielded the highest mechanical properties, with flexural strength reaching 370.65 MPa. The light transmission remained at 89%, equivalent to untreated glass, while achieving a potassium ion diffusion depth of 16 μm . Compared to the traditional salt bath method which achieved 575.61 MPa flexural strength, the spray coating technique achieved 370.65 MPa (approximately 64% of salt bath performance) while offering significant advantages including reduced potassium nitrate consumption, shorter processing times, and enhanced compatibility with complex geometries, making it a promising alternative for industrial glass strengthening applications.

Keywords: Chemical strengthening; Ion exchange; Glass coating; Kaolin slurry; Surface hardening

Introduction

Glass packaging, despite its chemical durability, aesthetic appeal, and strength, faces challenges in transportation due to its weight compared to other packaging materials [1]. While recent developments have produced thinner, lighter glass containers, this reduction in thickness compromises strength [2, 3]. The challenge lies in developing lightweight glass packaging with enhanced surface strength.

Chemical strengthening through ion exchange, particularly between Na^+ and K^+ ions at the glass

surface, has been established as an effective method for improving glass strength [4]. The larger K^+ ions (0.137 nm for 4-coordination) replacing smaller Na^+ ions (0.099 nm for 4-coordination) create compressive stress at the glass surface [5, 6]. This chemical strengthening approach offers advantages over thermal strengthening, particularly for thin glass (<2 mm thickness) [7]. While effective, these methods have limitations - thermal tempering requires uniform thickness, and salt bath immersion

is time-consuming and resource-intensive for industrial applications [8, 9].

Previous studies have explored chemical strengthening using spray methods with $\text{KNO}_3\text{-Al}_2\text{O}_3$ solutions [10, 11]. This research explores an alternative approach combining spray coating and microwave heating to achieve glass strengthening. The technique uses a KNO_3 -calcined kaolin slurry spray coating followed by microwave heating, potentially offering faster processing times and reduced energy consumption compared to conventional methods [12, 13].

Materials and Methods

The experimental procedure began with the preparation of the potassium nitrate solution. A 21.5 wt% KNO_3 solution was prepared by dissolving 32.25g of potassium nitrate (KNO_3 , 99.4%, AR grade, destiny chemicals) in 150 mL of deionized water at room temperature for 4 hours under constant stirring. For the preparation of the KNO_3 -calcined kaolin slurry, kaolin clay was first sieved through a 200-mesh screen and calcined at 700°C for 30 minutes with a heating rate of 5°C per minute. The calcined kaolin (6.8 wt%, 10.2g) was then added to the prepared KNO_3 solution and ball-milled for 1 hour to ensure uniform dispersion. The slurry composition (21.5 wt% KNO_3 with 6.8 wt% calcined kaolin) was optimized based on preliminary trials to balance potassium ion availability with uniform coating application; the calcined kaolin serves as a carrier matrix that maintains intimate contact between the potassium source and glass surface during thermal treatment while preventing salt crystallization that could occur at higher KNO_3 concentrations.

Soda-lime glass slides (25.4 mm × 76.2 mm × 1.0 mm) were thoroughly cleaned using ethanol ($\text{C}_2\text{H}_6\text{O}$, 99.9%, Grade AR) and dried in an oven at

110°C for 1 hour. The cleaned glass slides were placed on a hot plate maintained at 130°C , and the prepared KNO_3 -calcined kaolin slurry was sprayed onto both sides of the glass using a hand spray gun. Two coating passes were made on each side to ensure uniform coverage. The coated samples were weighed to ensure consistency in coating thickness across all specimens.

The thermal treatment process was conducted using three different approaches. In the first approach, samples were heated in a microwave oven (SHARP R380I, 1200W) at either 40% or 50% power for 10 minutes. Two power levels (40% and 50%, corresponding to approximately 480W and 600W effective power) were selected to investigate the threshold energy required to initiate ion mobility while avoiding thermal damage to the glass structure; the 40% power level provides gentler heating that maintains structural integrity, while the 50% power level was tested to explore whether higher energy input could enhance ion exchange kinetics. The samples were placed in an alumina box coated with silicon carbide as a susceptor, which was positioned on an alumina fiber board with grooves to allow even heat distribution (as shown in **Figure 1**). In the second approach, samples were heated in an electric furnace at 470°C for various durations (1, 4, 8, and 16 hours). The third approach combined both methods, where samples were first treated in the microwave (40% or 50% power, 10 minutes) followed by electric furnace treatment (470°C for 1, 1.5, or 4 hours).

For comparison purposes, additional samples were treated using the traditional salt bath method, where glass slides were immersed in molten KNO_3 at 470°C for 1.5 and 4 hours. After all thermal treatments, the samples were allowed to cool naturally to room temperature inside their respective heating chambers to prevent thermal shock.

Following the cooling period, all treated samples were thoroughly cleaned with deionized water and ethanol to remove any residual coating or salt.

The samples were then subjected to various characterization techniques. Mechanical properties were evaluated using a three-point bending test with a 40 mm span and 5 mm/min loading rate, and Vickers microhardness testing with a 5 kgf load applied for 15 seconds. For the hardness measurements, indentations were spaced at least 3-5 times the crack length apart to prevent interference between stress fields. The mechanical strengthening was evaluated through flexural strength and surface hardness measurements, which serve as indirect

indicators of the compressive stress layer generated by ion exchange; direct measurement of residual compressive stress using optical methods such as scattered light polariscopy (SCALP) is recommended for future studies to provide quantitative stress profile data. Optical transmission was measured using a UV-VIS spectrophotometer over the visible wavelength range (380-780 nm). The ion exchange depth and distribution were analyzed using SEM-EDS line scanning, with samples prepared by cross-sectioning and polishing. For each treatment condition, ten samples were tested to ensure statistical reliability of the results.

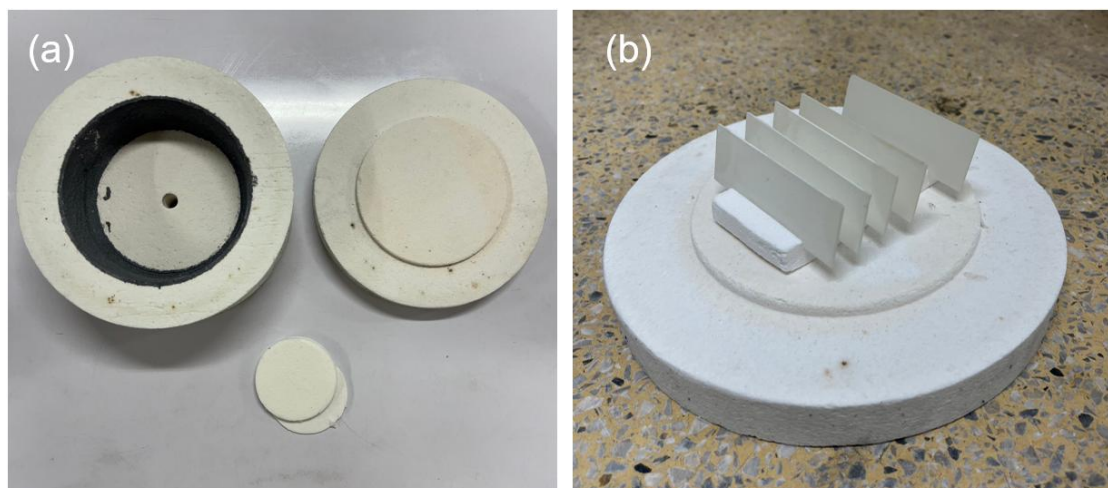


Figure 1. (a) an alumina box coated with silicon carbide as a susceptor

(b) Glass slides sprayed with KNO_3 + calcined kaolin slurry and placed on alumina support

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Results and discussion

1. Effect of Microwave Treatment

1.1 Mechanical Properties

Samples treated with microwave heating alone showed varying degrees of improvement in mechanical properties as shown in **Figure 2**. At 40% microwave power for 10 minutes, the flexural strength reached 192.58 MPa, comparable to the untreated glass (192.38 MPa). However, increasing the power to 50% resulted in a slight decrease to 181.27 MPa. The decrease in flexural strength at higher microwave power (50%) is attributed to non-uniform temperature distributions causing localized thermal stresses and partial stress relaxation at the glass surface, which counteract the ion exchange strengthening mechanism. At higher power levels, more rapid and localized heating creates temperature gradients that induce thermal stresses rather than the beneficial compressive stresses generated by ion exchange. This minimal change in flexural strength suggests that microwave treatment alone is insufficient for significant mechanical property enhancement.

The surface hardness measurements (**Figure 3**)

revealed more promising results. Samples treated at 50% microwave power showed the highest hardness value of 543.27 HV, while 40% power treatment achieved 530.52 HV, both showing improvement over the untreated glass (511.05 HV). This differential between flexural strength and hardness improvements indicates that microwave treatment primarily affects the surface properties rather than the bulk material.

1.2 Ion Exchange Characteristics

SEM-EDS analysis revealed that samples treated at 50% microwave power exhibited a K^+ ion diffusion depth of 8 μm (**Figure 4**), while no significant ion penetration was detected at 40% power. This observation suggests that a threshold power level is necessary to initiate the ion exchange process. The limited diffusion depth explains the modest improvements in mechanical properties, as effective chemical strengthening typically requires deeper ion penetration.

The EDS linescan profiles are superimposed on the cross-sectional SEM image, where the red line represents Na signal intensity and the cyan line represents K signal intensity. The vertical axis indicates relative intensity in arbitrary units. The horizontal direction corresponds to spatial position from bulk glass (left) toward the surface (right), with the white arrow indicating the glass surface location. The K^+ ion diffusion depth of approximately 8 μm is determined from the position where K intensity decreases to the baseline level.

2 Effect of Electric Furnace Treatment

2.1 Mechanical Properties

Electric furnace treatment showed a clear time-dependent effect on mechanical properties (**Figure 5**). The highest flexural strength (319.83 MPa) was achieved after 1 hour at 470°C, followed by a gradual decrease with longer treatment times: 268.90 MPa at 4 hours, 255.52 MPa at 8 hours, and

196.38 MPa at 16 hours. This trend suggests that extended heat treatment may lead to stress relaxation in the glass structure, potentially compromising the strengthening effect.

Surface hardness measurements showed an opposite trend, with values increasing with treatment

time (**Figure 6**). The maximum hardness of 564.40 HV was achieved after 16 hours, significantly higher than the untreated glass. This contrasting behavior between flexural strength and hardness indicates different mechanisms affecting surface and bulk properties during thermal treatment.

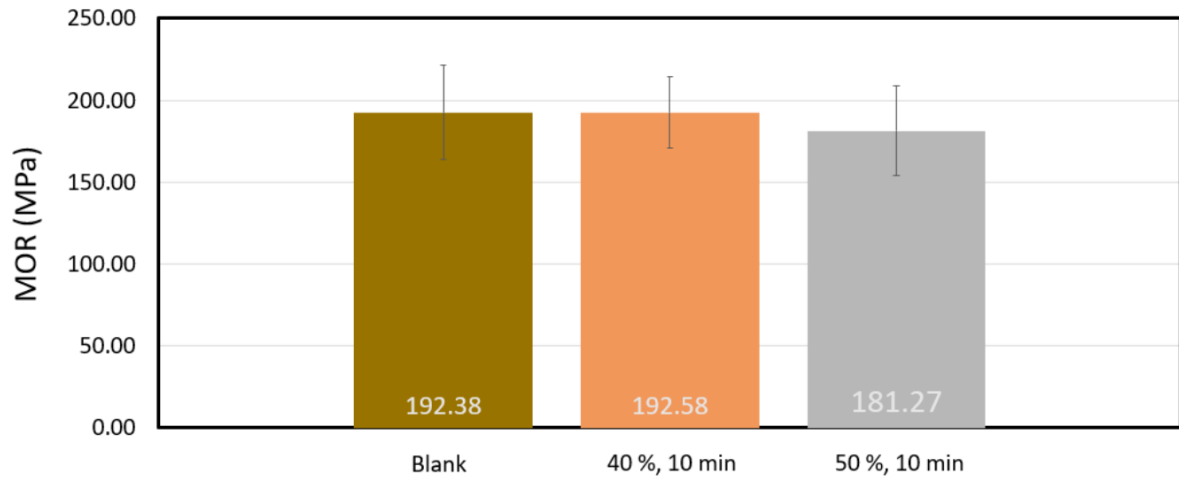


Figure 2. Flexural strength of glass samples after spray coating with KNO_3 – calcined kaolin slurry and microwave heat treatment at 40% and 50% power for 10 minutes, respectively.

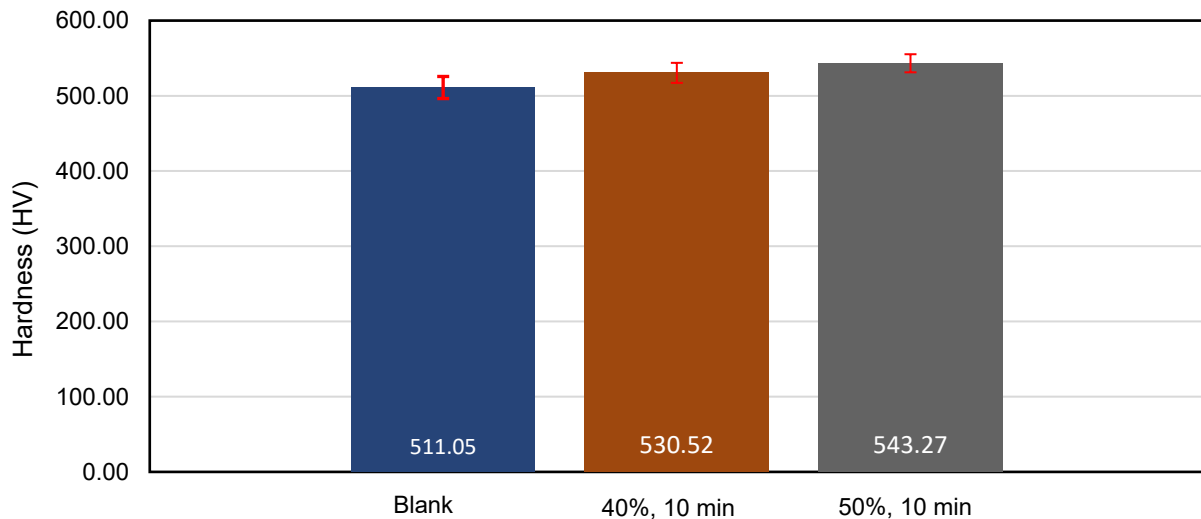


Figure 3. Surface hardness of glass samples after spray coating with KNO_3 – calcined kaolin slurry and microwave heat treatment at 40% and 50% power for 10 minutes, respectively.

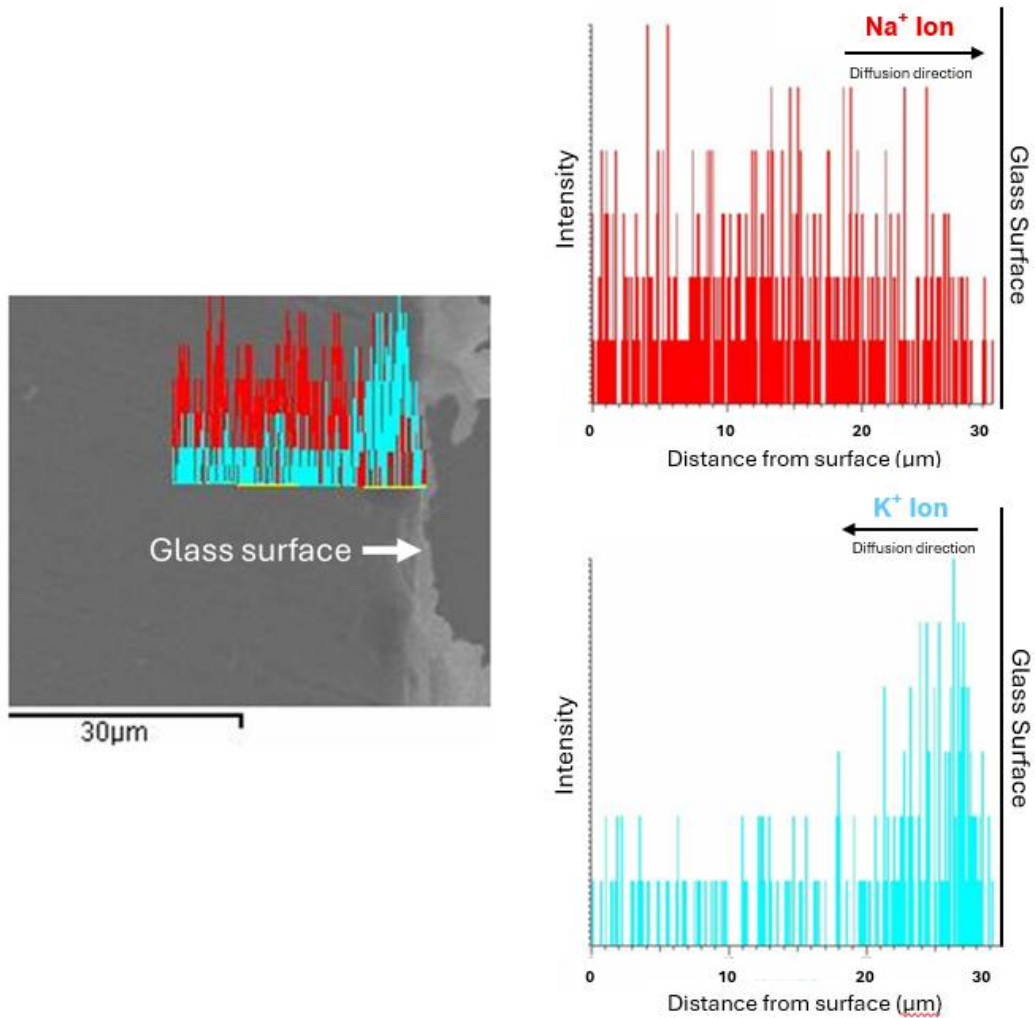


Figure 4. SEM micrograph with overlaid EDS linescan analysis of glass sample after spray coating with KNO_3 -calcined kaolin slurry and microwave heat treatment at 50% power for 10 minutes.

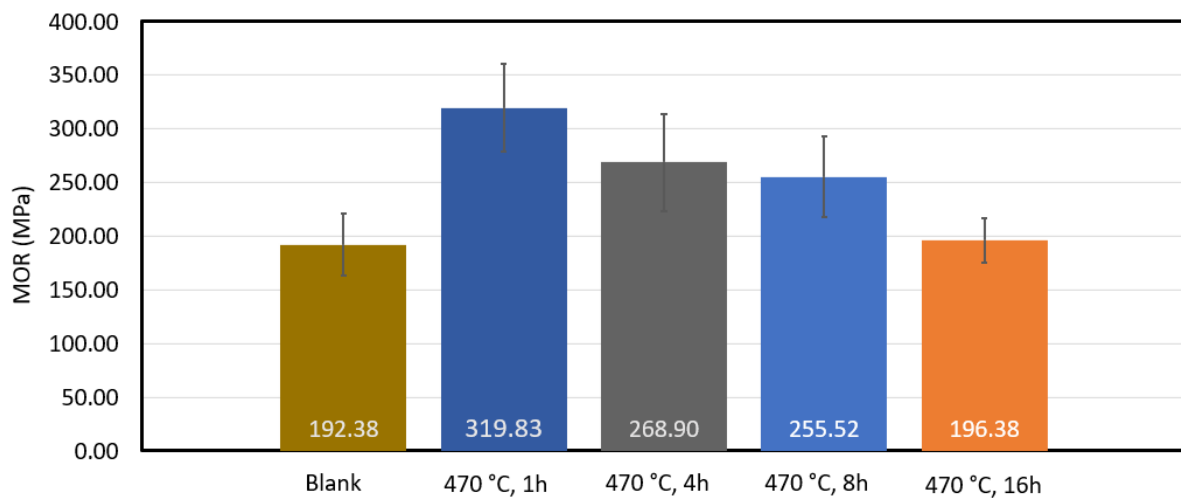


Figure 5. Flexural strength of glass samples after spray coating with KNO_3 – calcined kaolin slurry and electric furnace heat treatment at 470 °C for 1, 4, 8, and 16 hours, respectively.

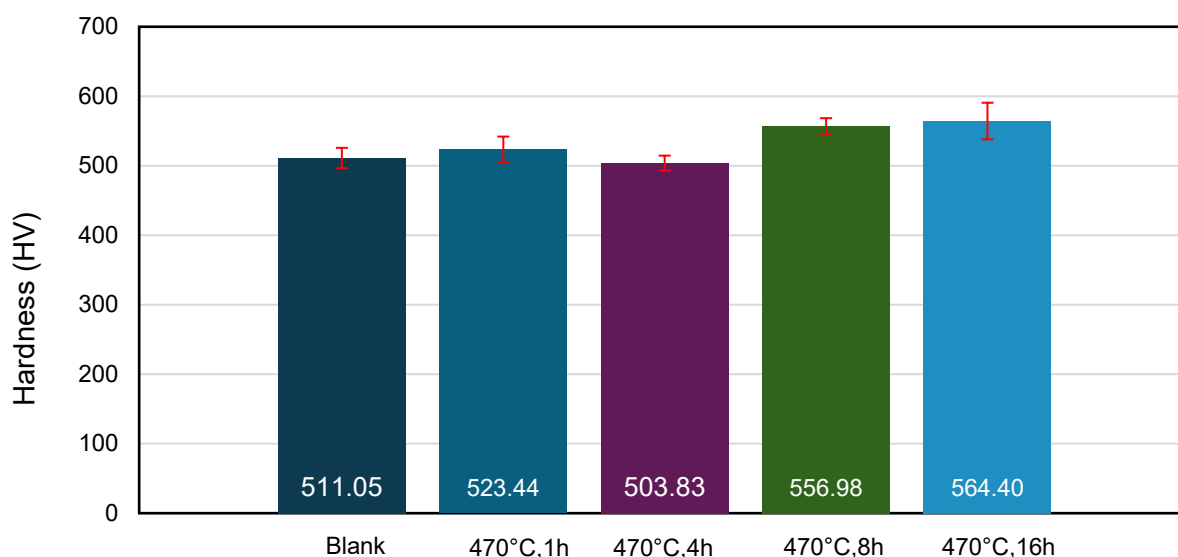


Figure 6. Surface hardness of glass samples after spray coating with KNO_3 – calcined kaolin slurry and electric furnace heat treatment at 470 °C for 1, 4, 8, and 16 hours, respectively.

2.2 Ion Exchange Depth

The ion exchange depth (**Figure 7**) increased progressively with treatment time, reaching 18 μm after 16 hours, 17 μm after 8 hours, and 11 μm after 4 hours. The gradual increase in diffusion depth correlates well with the observed hardness improvements but suggests that longer treatment times may lead to a more uniform stress distribution, explaining the decrease in flexural strength.

3 Combined Microwave and Furnace Treatment

3.1 Mechanical Properties

The combination of microwave pre-treatment followed by furnace heating proved most effective. Samples treated with 40% microwave power for 10 minutes followed by 4 hours at 470°C achieved the highest flexural strength of 370.65 MPa (**Figure 8**). This represents a 92.7% increase over the untreated glass and is significantly higher than either treatment method alone. The synergistic effect suggests that microwave pre-treatment may enhance subsequent ion exchange during furnace heating.

Surface hardness values (as shown in **Figure 9**) for the combined treatment remained consistently

high across all conditions (537.80-562.11 HV), with minimal variation between different treatment parameters. This consistency indicates that the combined treatment provides reliable surface strengthening regardless of specific power levels or furnace duration.

3.2 Ion Exchange Characteristics and Optical Properties

The combined treatment achieved a maximum K^+ ion diffusion depth of 16 μm under optimal conditions (40% microwave power, 10 minutes + 470°C, 4 hours) (**Figure 10 - 11**). This substantial diffusion depth, coupled with maintained optical transparency (89% transmission) as shown in **Figure 12**, demonstrates the process's effectiveness in achieving strengthening without compromising optical properties.

The EDS linescan profiles are superimposed directly on the cross-sectional SEM image to show the spatial correlation between microstructure and elemental distribution. The red line represents $\text{Na K}\alpha$ intensity and the cyan line represents $\text{K K}\alpha$ intensity, with the vertical axis indicating relative intensity in arbitrary units. The linescan direction

proceeds from the bulk glass (left side) toward the surface (right side), with the white arrow marking the glass surface position. The scale bar represents 30 μm . The K-enriched zone is clearly visible as the region of high cyan (K) signal intensity near the glass surface, extending approximately 16 μm into the glass, while the Na signal (red) shows corresponding depletion in this ion-exchanged region. This overlay presentation allows direct visualization of how the K^+ penetration depth correlates with the glass cross-sectional morphology.

The effect of slurry content on ion exchange efficiency warrants discussion. The KNO_3 in the slurry serves as the source of K^+ ions for the exchange process, while the calcined kaolin matrix facilitates uniform delivery of these ions to the glass surface during thermal treatment. Increasing the KNO_3 concentration beyond the current 21.5 wt% could potentially enhance K^+ ion availability and increase the diffusion depth. However, preliminary trials indicated that higher KNO_3 concentrations led to crystallization during the spray coating process, resulting in non-uniform coverage that negatively affected the mechanical properties. The current slurry composition represents an optimized balance between K^+ availability and coating uniformity. A comprehensive summary of mechanical properties, ion diffusion depth, and light transmission for all treatment conditions is presented in Table 1.

4 Comparison with Traditional Salt Bath Method

The traditional salt bath method (470°C, 1.5 hours) achieved the highest overall flexural strength of 575.61 MPa and surface hardness of 593.09 HV (**Figure 13-14**). However, this method requires significantly more potassium nitrate and longer processing times compared to the spray coating technique. The optical properties of the glass remained equivalent across all methods after

cleaning, with ~89% light transmission maintained. (**Figure 15**).

The comparison between salt bath and combined microwave-furnace treatment methods reveals important practical tradeoffs in manufacturing efficiency. While the salt bath approach demonstrated superior mechanical properties, the microwave-furnace treatment with spray coating presents compelling advantages from an operational perspective. This alternative method significantly reduces material consumption and energy requirements, leading to more sustainable manufacturing practices. Processing times are notably shortened, allowing for higher throughput in production lines. The technique also shows greater versatility when handling complex geometries, eliminating some of the limitations inherent in salt bath treatments. Furthermore, its compatibility with existing manufacturing infrastructure makes implementation more straightforward, reducing the need for extensive facility modifications. These practical benefits make the microwave-furnace approach an attractive option despite the slight compromise in mechanical properties.

The investigation into strengthening mechanisms reveals a complex interplay of physical and chemical processes contributing to the enhanced glass properties. At the core of the strengthening process is an ion exchange mechanism where smaller sodium ions (0.95 Å) are replaced by larger potassium ions (1.33 Å), generating a beneficial compressive stress layer at the glass surface - a phenomenon clearly documented through SEM-EDS depth profiling. While the compressive stress was not directly measured using optical methods such as scattered light polariscopy (SCALP), the significant improvement in flexural strength (from 192.38 MPa to 370.65 MPa) provides strong indirect evidence of effective compressive stress generation,

as the glass must overcome this surface compression before tensile failure can initiate. This primary mechanism is complemented by thermal effects from the microwave treatment phase, which appears to establish advantageous stress distributions that optimize the subsequent ion exchange process during furnace treatment. Surface protection and ion delivery are simultaneously achieved through the application of calcined kaolin, which serves as an effective potassium ion carrier

while maintaining the glass's optical integrity during heat exposure. The success of this combined approach can be attributed to the microwave pre-treatment's unique ability to enhance ion mobility and create optimal conditions for the following ion exchange phase during furnace treatment. Critical to this process is the precise control of parameters to prevent stress relaxation, highlighting the importance of maintaining specific processing conditions throughout the treatment sequence

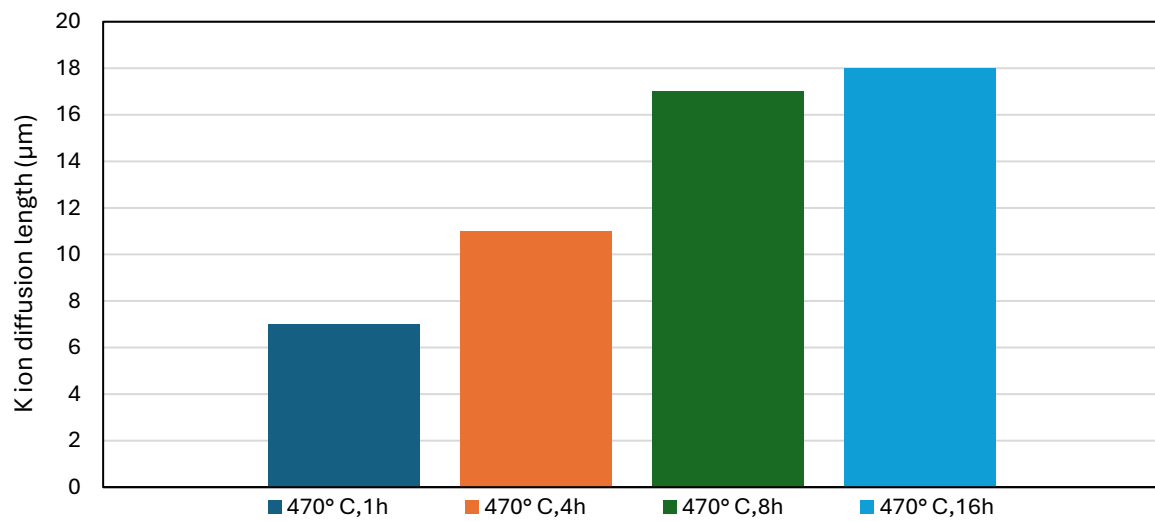


Figure 7. K⁺ ion diffusion depth in glass samples after spray coating with KNO₃ – calcined kaolin slurry and electric furnace heat treatment at 470 °C for 1, 4, 8, and 16 hours, respectively.

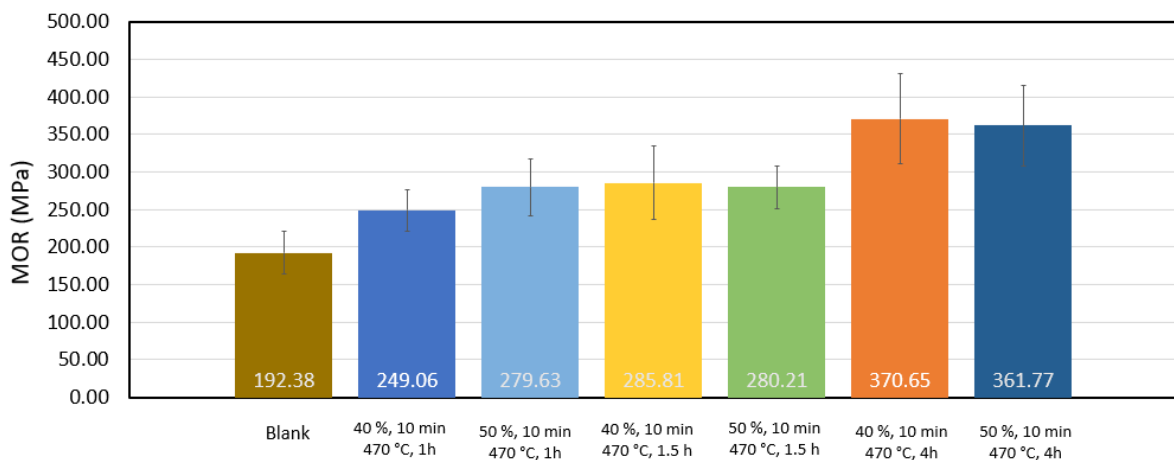


Figure 8. Flexural strength of glass samples after spray coating with KNO₃ – calcined kaolin slurry, subjected to a two-step heat treatment: initial microwave heating at 40% and 50% power for 10 minutes, followed by electric furnace treatment at 470 °C for 1, 1.5, and 4 hours, respectively.

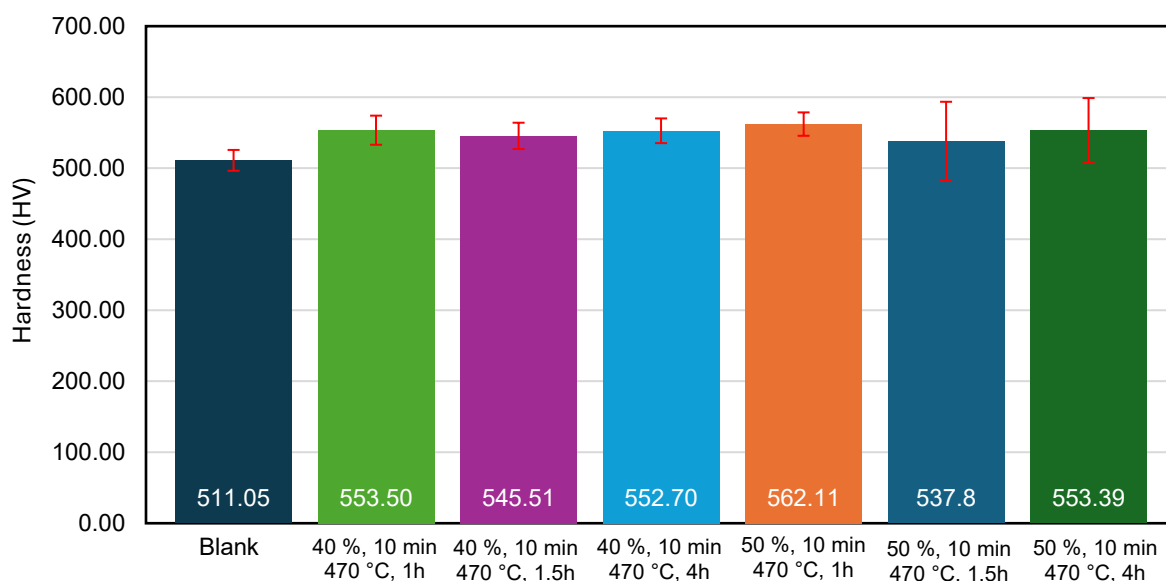


Figure 9. Surface hardness of glass samples after spray coating with KNO_3 – calcined kaolin slurry, subjected to a two-step heat treatment: initial microwave heating at 40% and 50% power for 10 minutes, followed by electric furnace treatment at 470 °C for 1, 1.5, and 4 hours, respectively.

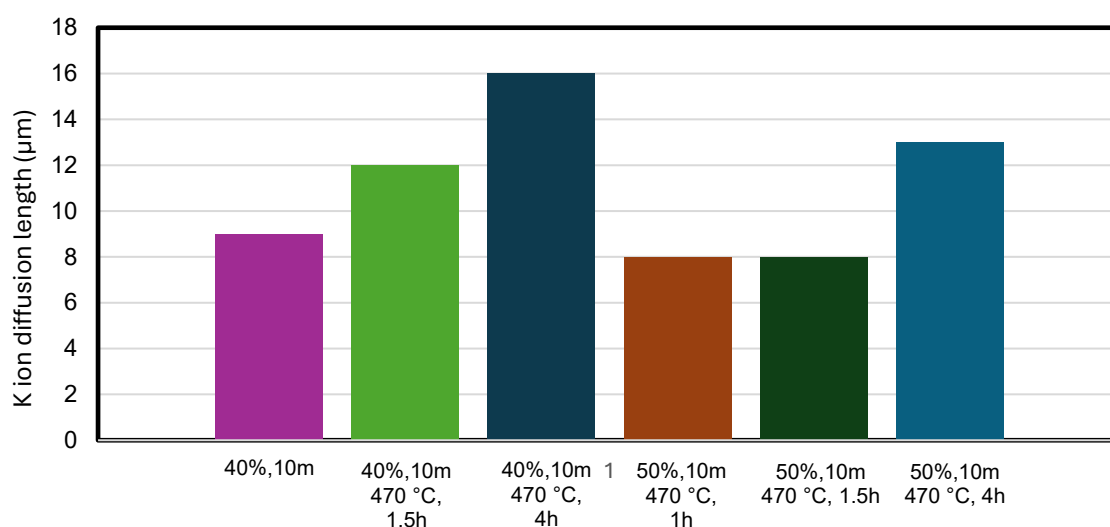


Figure 10. K^+ ion diffusion depth in glass samples after spray coating with KNO_3 – calcined kaolin slurry, subjected to a two-step heat treatment: initial microwave heating at 40% and 50% power for 10 minutes, followed by electric furnace treatment at 470 °C for 1, 1.5, and 4 hours, respectively.

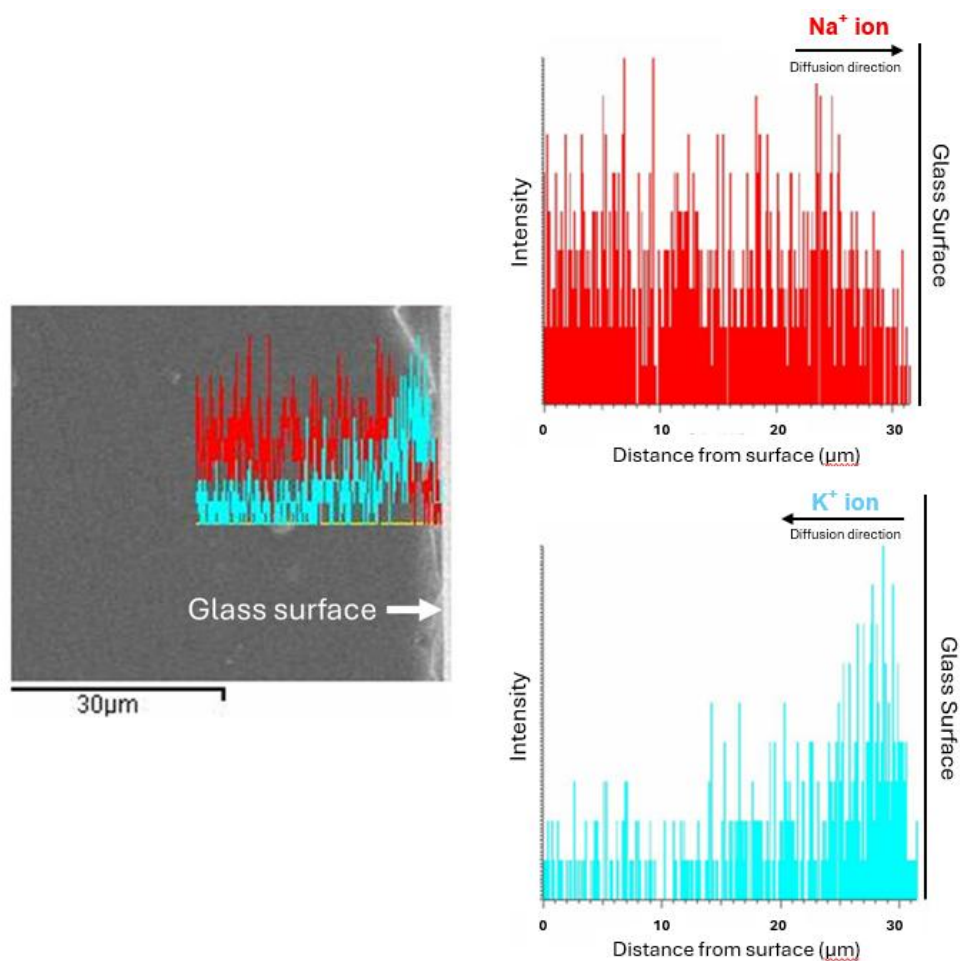


Figure 11. SEM micrograph with overlaid EDS linescan analysis of glass sample after spray coating with KNO_3 -calcined kaolin slurry, subjected to two-step heat treatment: initial microwave heating at 40% power for 10 minutes, followed by electric furnace treatment at 470°C for 4 hours.

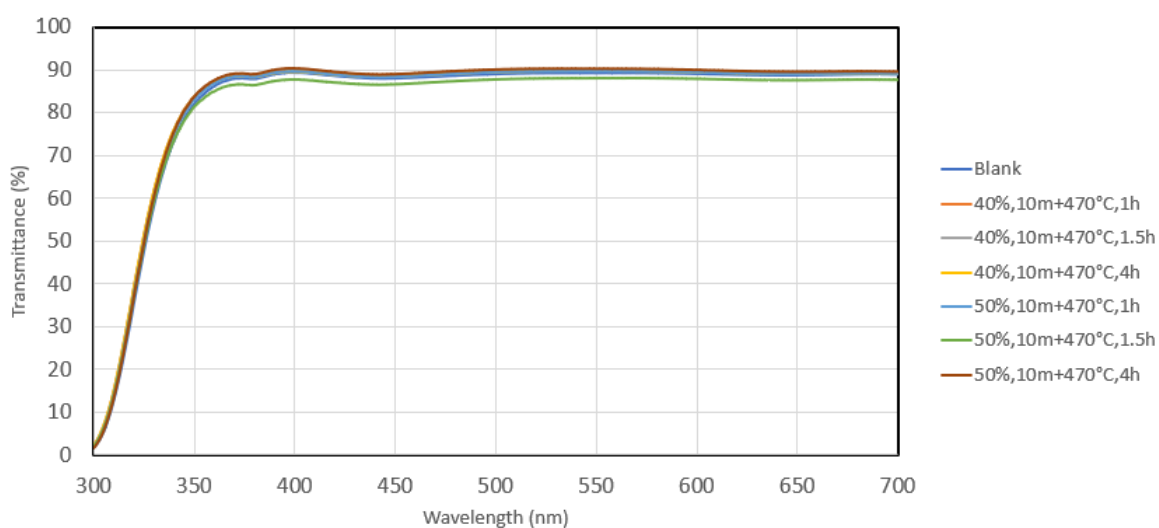


Figure 12. Light transmission of glass samples after spray coating with KNO_3 – calcined kaolin slurry, subjected to a two-step heat treatment: initial microwave heating at 40% and 50% power for 10 minutes, followed by electric furnace treatment at 470 °C for 1, 1.5, and 4 hours, respectively.

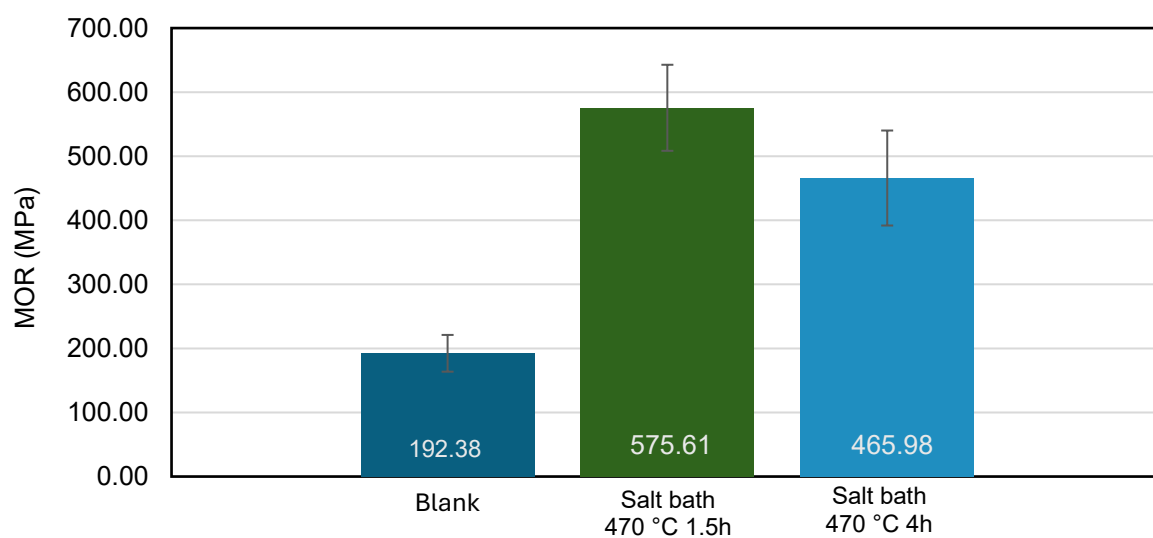


Figure 13. Flexural strength of ion-exchanged microscope slide glass in KNO_3 molten salt at 470 °C for 1.5 and 4 h

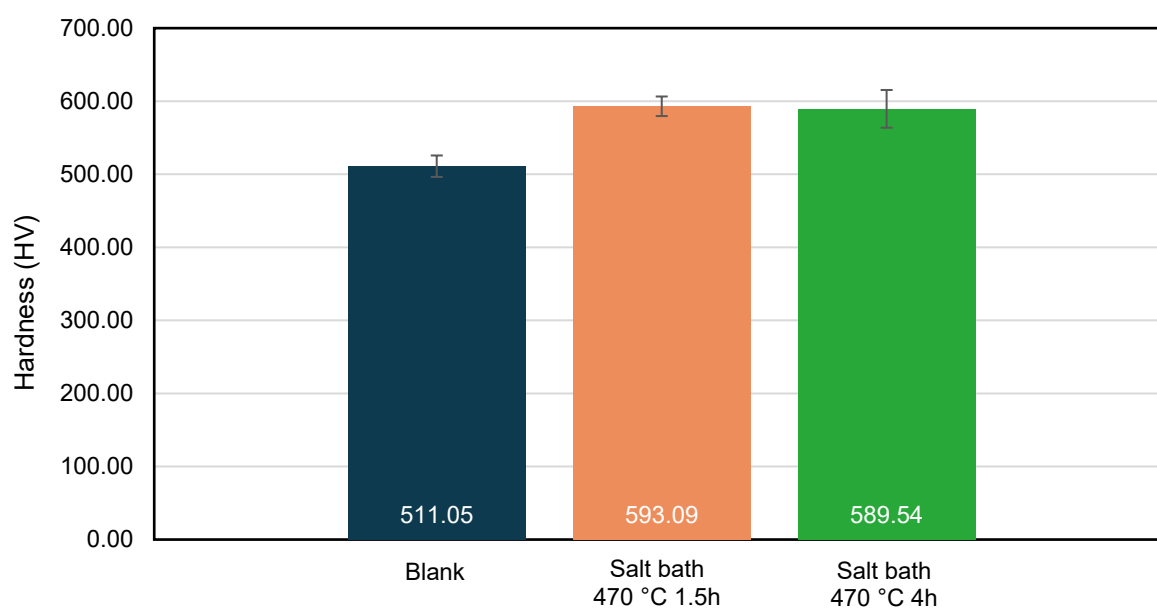


Figure 14. Surface hardness of ion-exchanged microscope slide glass in KNO_3 molten salt at 470 °C for 1.5 and 4 h

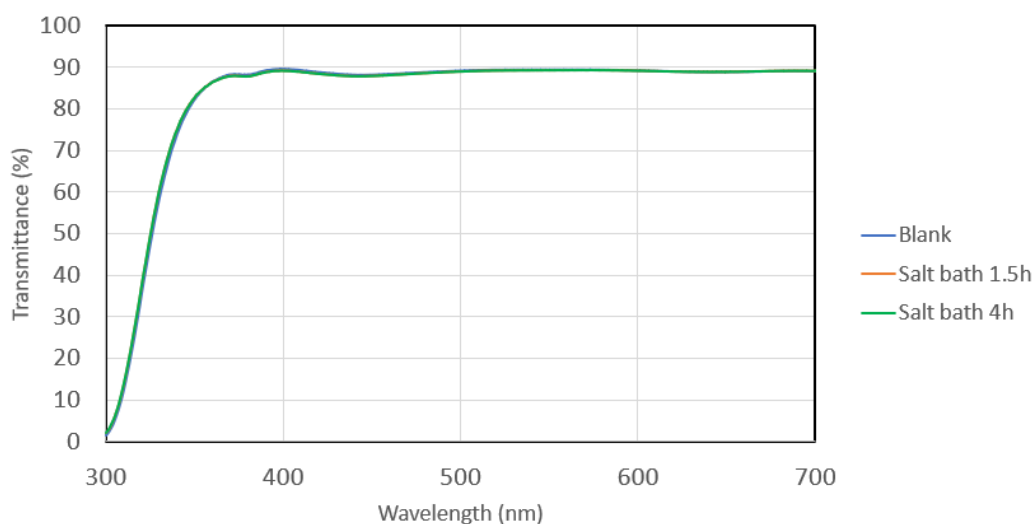


Figure 15. Light transmission of glass samples after immersion in KNO_3 salt bath at 470°C for 1.5 and 4 hours, respectively."

Table 1. Summary of mechanical properties, ion diffusion depth, and light transmission for all treatment conditions

Treatment Condition	Flexural Strength (MPa)	Hardness (HV)	K ⁺ Diffusion (μm)	Transmission (%)
Untreated (Blank)	192.38	511.05	-	89
MW 40%, 10 min	192.58	530.52	-	-
MW 50%, 10 min	181.27	543.27	8	-
Furnace 470°C , 1h	319.83	523.44	-	-
Furnace 470°C , 4h	268.90	503.83	11	-
Furnace 470°C , 8h	255.52	556.98	17	-
Furnace 470°C , 16h	196.38	564.40	18	-
MW 40% + 470°C , 1h	249.06	553.50	-	~89
MW 40% + 470°C , 1.5h	285.81	545.51	-	~89
MW 40% + 470°C , 4h	370.65	552.70	16	89
MW 50% + 470°C , 1h	279.63	562.11	-	~89
MW 50% + 470°C , 1.5h	280.21	537.80	-	~89
MW 50% + 470°C , 4h	361.77	553.39	-	~89
Salt bath 470°C , 1.5h	575.61	593.09	-	~89
Salt bath 470°C , 4h	465.98	589.54	-	~89

Conclusion

This study demonstrates the effectiveness of combining microwave heating with conventional furnace treatment for glass strengthening. The process achieves significant improvements in mechanical properties while maintaining optical transparency. Key findings include:

- 1) Optimal treatment conditions: 40% microwave power (10 minutes) + 470°C furnace treatment (4 hours)

- 2) Maximum flexural strength: 370.65 MPa
- 3) Preserved optical transmission: 89%
- 4) Ion diffusion depth: 16 μm

The results suggest that this hybrid approach could offer an energy-efficient alternative to traditional strengthening methods, particularly for industrial applications requiring rapid processing times. Future work should include direct measurement of residual compressive stress profiles using optical techniques to further quantify the strengthening mechanism.

Acknowledgements)

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