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Investigation of the Effects of Mechanical Washing Resistance and Weather Conditions on Chemically Strengthened Crystal Glasses

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ABSTRACT

The chemical tempering method is a versatile process applicable to glass of various sizes, complex shapes, and thin structures (up to 0.5 mm). Evaluating chemical strengthening methods under different weather conditions is crucial for developing durable glassware. This study examines the impact of mechanical washing resistance and weather conditions on untreated and chemically tempered glass through experimental analysis. Tempered samples exhibit an average Compressive Stress (CS) of 413 MPa (±8 MPa), a Layer Depth (DOL) averaging 20 µm (±2 µm), and Central Stress (CT) averaging 9 MPa (±0.6 MPa). Despite this, Energy Dispersion Spectroscopy (EDS) images show no visible impact on surface morphology post-tempering. Nevertheless, EDS spectra indicate an increased potassium ratio, confirming successful chemical tempering. The hardness of chemically tempered glass is 6.16 GPa (±0.18), significantly higher than untempered glass at 5.53 GPa (±0.12). In the Free Fall Test, untempered glass drops an average of 16 cm, while tempered glass surpasses 28 cm without breaking. During the Bending Test, untempered glass bends 6 degrees on average, whereas tempered glass exceeds 13 degrees without breaking. Chemically tempered glass resists hydrolysis well and shows no significant difference in dishwasher resistance, but it excels in breakage resistance under test conditions.

Mekanik Bulaşık Yıkama Direncinin ve Hava Şartlarının Kimyasal Olarak Güçlendirilmiş Kristal Camlar Üzerindeki Etkisinin İncelenmesi

ÖZ

Kimyasal temperleme yöntemi, çeşitli boyutlarda, karmaşık şekillerde ve ince yapılardaki (0,5 mm'ye kadar) camlara uygulanabilen çok yönlü bir işlemdir. Farklı hava koşullarında kimyasal güçlendirme yöntemleri, dayanıklı cam eşya geliştirmek için çok önemlidir. Bu çalışma, mekanik yıkama direnci ve hava koşullarının işlenmemiş ve kimyasal olarak temperlenmiş cam üzerindeki etkisini deneysel analiz yoluyla incelemektedir. Temperlenmiş numuneler ortalama 413 MPa (±8 MPa) Basınç Gerilimi (CS), ortalama 20 µm (±2 µm) Katman Derinliği (DOL) ve ortalama 9 MPa (±0,6 MPa) Merkezi Gerilim (CT) sergiler. Buna rağmen Enerji Dağılımlı X-ışını Spektroskopisi (EDS) görüntüleri yüzey morfolojisi sonradan temperleme üzerinde gözle görülür bir etki göstermemektedir. Bununla birlikte, EDS spektrumları, başarılı kimyasal tavlamayı doğrulayan artan bir potasyum oranına işaret etmektedir. Kimyasal olarak temperlenmiş camın sertliği 6,16 GPa (±0,18) olup, 5,53 GPa (±0,12) ile temperlenmemiş camdan önemli ölçüde daha yüksektir. Serbest Düşme Testinde temperlenmemiş cam ortalama 16 cm düşerken, temperli cam 28 cm'yi kırılmadan aşar. Eğilme Testi sırasında temperlenmemiş cam ortalama 6 derece bükülürken, temperli cam kırılmadan 13 dereceyi aşar. Kimyasal olarak temperlenmiş cam, hidrolize karşı iyi direnç gösterir ve bulaşık makinesinde önemli bir fark göstermez, ancak test koşullarında kırılma direncinde üstünlük sağlar.

Anahtar Kelimeler: Kimyasal temperli cam , Bulaşık Makinesinde Yıkanabilme Dayanımı , Hava Şartları

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1. Introduction

Commonly perceived as a solid substance, glass actually exhibits a distinct nature as an amorphous material formed through rapid cooling with high viscosity, preventing crystallization. Essentially, it is a semi-ordered substance, lacking the repeating and organized patterns found in crystalline materials [1]. The American International Society for Testing and Materials (ASTM) provides a widely accepted definition of glass: "an inorganic product of fusion that has cooled to a rigid condition without crystallizing; a noncrystalline solid or an amorphous solid." This definition emphasizes the non-crystalline nature of glass [2].

Glass, a material with a rich history dating back thousands of years, has evolved significantly in its composition and applications. Glass has found many applications over the years and has gained a place in the construction industry, architecture, defense industry, household goods, and many other fields. Its wide usage area has led to the production and use of glass with new techniques [3-5]. Glass generally exhibits high mechanical strength and chemical resilience. However, its tensile strength is relatively low, a characteristic that can be attributed to the presence of surface defects known as cracks. These cracks significantly impact the overall mechanical properties of glass, affecting its strength and durability [6]. This vulnerability is especially noticeable when exposed to water vapor and temperature variations, leading to atmospheric corrosion known as "weathering" or "warehouse effect." Consequently, these surface changes exacerbate the impact of cracks, collectively shaping the material's overall resilience and long-term performance. In conditions of high humidity, there is a gradual expansion of cracks over time. This mechanicochemical process negatively impacts the mechanical and optical characteristics of glass, influencing properties such as fracture strength [7], surface roughness [8], and transmittance [9].

The primary corrosion processes can occur when silica-based glasses are exposed to an aqueous environment, ion exchange reaction takes place between the modifier ions of the glass network and hydrous species of water. This mechanism is commonly referred to as leaching [10]. The rate-determining step in leaching reactions can be described as follows;

$$Si - O - \ M^{+} + \ H_2O \rightarrow Si - OH + OH^{-} + \ M^{+} \qquad (1)$$

where M represents the alkali ions present in the silicate glass network. This reaction illustrates the primary corrosion mechanism involving the ion exchange process between metal ions within the glass and H+ ions from the aqueous solution. The interaction of hydrogen with oxygen may result in condensation, and the subsequent evaporation of water can trigger cracking on the surface of the glass [8]. The rate at which these cracks expand is affected by several factors, such as humidity [10], temperature, applied stress, time, and the composition of the glass [11].

Glass often undergoes repeated exposure to humidity and various weather conditions throughout its lifespan. An illustrative example is the dishwashing process. Through the long-term dishwashing process, the glass interacts with water and detergents at elevated temperatures and for extended durations compared to manual handwashing. This exposure can lead to irreversible surface degradation, such as white clouding, iridescence, and other optical alterations on the glass surface. Numerous research papers have extensively investigated the damage to glass surfaces caused by different dishwashing detergents, varying washing temperatures, and distinct water hardness levels [12,13].

Therefore, enhancing the strength of glass is crucial, and various methods for glass strengthening have been extensively developed in recent decades. These methods include thermal tempering [14], chemical strengthening [15-17], and surface crystallization [18].

There is a growing interest in chemical tempering, also known as ion exchange or chemical strengthening, due to its excellent mechanical properties [19].

Chemical strengthening is achieved essentially by replacing smaller ions in the glass surface with larger ions. without measurable optical distortion, making it the leading candidate for enhancing glass strength [20]. The chemical tempering method is versatile and can be easily applied to glass sections of different sizes, complex shapes, and very thin structures (up to 0.5 mm). It has advantages over other forms of glass strengthening, such as thermal tempering, as it can produce thinner and lighter glass with similar or better strength characteristics [21].

In the chemical tempering process, ion exchange occurs between alkaline ions present in the glass structure and ions in a molten salt bath that is in contact with the glass surface. The smaller radius ions in the glass structure are replaced by larger ions from the molten salt, causing local strain. This, in turn, creates compressive stress on the glass surface. The compressive pressure resulting from the ion exchange strengthens the glass, making it stronger and more resistant to mechanical and thermal stress compared to untempered glass.

By evaluating the effectiveness of chemical strengthening methods for glass in real-world scenarios, more durable glassware that can withstand harsh environments for extended periods can be developed. Therefore, this study investigates the impact of mechanical dishwasher resistance and weather conditions on untempered and chemically tempered glass through experimental analysis. It measures the compressive stress level and stress layer depth, analyzes surface characteristics and composition, measures hardness, determines main oxide and trace element content as well as heavy metal content, and evaluates impact resistance, free fall resistance, and bending resistance of the glass products.

2. Materials and Strengthening Process

The molten salt used was pure KNO_3 (purity > 99.9%). The glass surfaces were carefully cleaned with pure deionized water. The FSM-6000LE from Lueco, Japan, was utilized to determine compressive stress levels and analyze the depth of the stress layer. Surface characteristics and composition analysis were conducted using a Zeiss Supra 40 VP model Field Emission Scanning Electron Microscope (SEM) equipped with an Energy Dispersion Spectroscopy (EDS) unit. To assess sample hardness, the Vickers hardness measurement methodology was applied using the Shimadzu Model-M Micro-rigidity, and the obtained hardness values were analyzed using the optical profiler Bruker Counter GT-K1. The principal oxide, trace element, and heavy metal content of the samples were analyzed through X-ray fluorescence (XRF) analysis.

The samples were placed within a containment basket to facilitate their introduction into the tempering process. Subsequently, subjecting the samples to a preliminary heating phase, exposing them to elevated temperatures. Compressive stress was induced on their surfaces when they were immersed in molten potassium nitrate salt during this preheating stage. After extraction from the salt bath, a meticulously controlled and gradual cooling process was applied, playing an important role in establishing the desired compressive stress within the crystalline structure. Thorough rinsing followed for the tempered samples, and their compliance with predetermined standards was evaluated. This systematic evaluation ensured the attainment of the targeted mechanical and chemical properties. Figure 1 illustrates a schematic of the chemical tempering process.

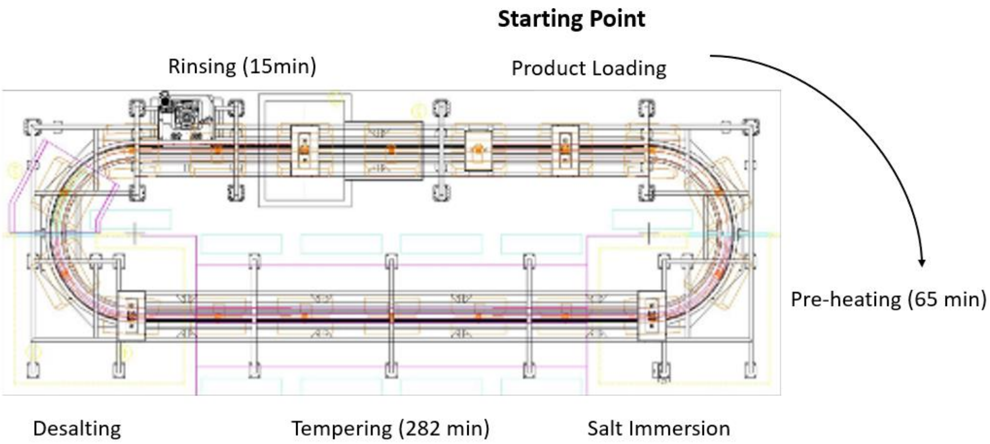

Figure 1. Chemical tempering line (Şişecam) (Patent No. 2018/01566).

3. Results and Discussion

3.1. FSM-6000LE analysis

The assessment of compressive stress (CS), layer depth (DOL), and central stress (CT) in two glass samples was conducted after chemical annealing. The average values were calculated based on five measurements for each glass sample, as shown in Table 1.

Table 1. Compressive stress, layer depth, and central stress values.

	Glass sample 1			Glass Sample 2		
Measurement No:	**Compressive stress (MPa)**	**Depth of layer (μm)**	**Central Tension (MPa)**	**Compressive stress (MPa)**	**Depth of layer (μm)**	**Central Tension (MPa)**
1	428	17	9	420	18	10
2	407	20	10	431	19	10
3	409	23	10	418	19	10
4	407	18	9	433	20	10
5	414	19	9	425	20	10
Average	413	20	9	425	19	10
Std. dev.	8	2	0,6	6	0,7	0,3

3.2. Surface morphology analysis

A thin conductive layer comprising 80% gold and 20% palladium was applied using the sputtering technique. Images were taken under a vacuum of 10-6 torr and with a 20 kV accelerating voltage.

Figure 2 shows SEM images of tempered and untempered glasses (both bulk and powder) taken at different magnifications. SEM images reveal that the tempering process does not visibly affect the surface morphology of the glass samples. The superficial nature of the tempering process suggests that changes in potassium and sodium did not impact the overall density of the glass.

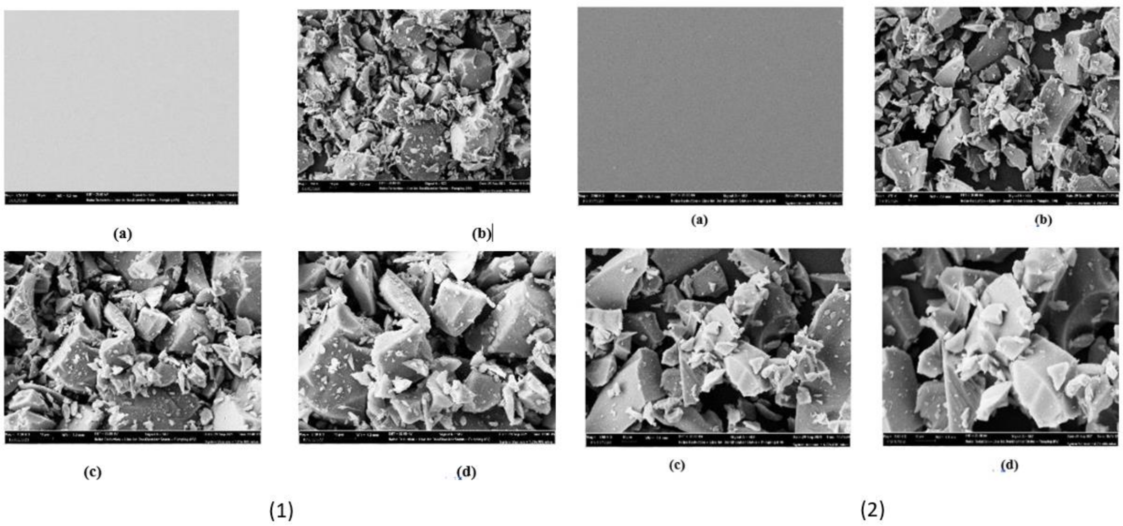

Figure 2. SEM images of tempered (1) and untampered (2) glasses (both bulk and powder) that were taken at different magnifications. a) bulk x2500 b) powder x750 c) powder x1500 and d) powder x2500.

Both untempered and tempered bulk glasses share the same matrix, yet there is a significant increase in the potassium ratio on the surface after tempering, as seen in Figure 3 and Figure 4. The tempering process is successful based on the analysis of surface morphology and composition. The differences observed in Figure 5 and Figure 6 in the potassium ratio between bulk and powder samples suggest that the tempering process is applied superficially.

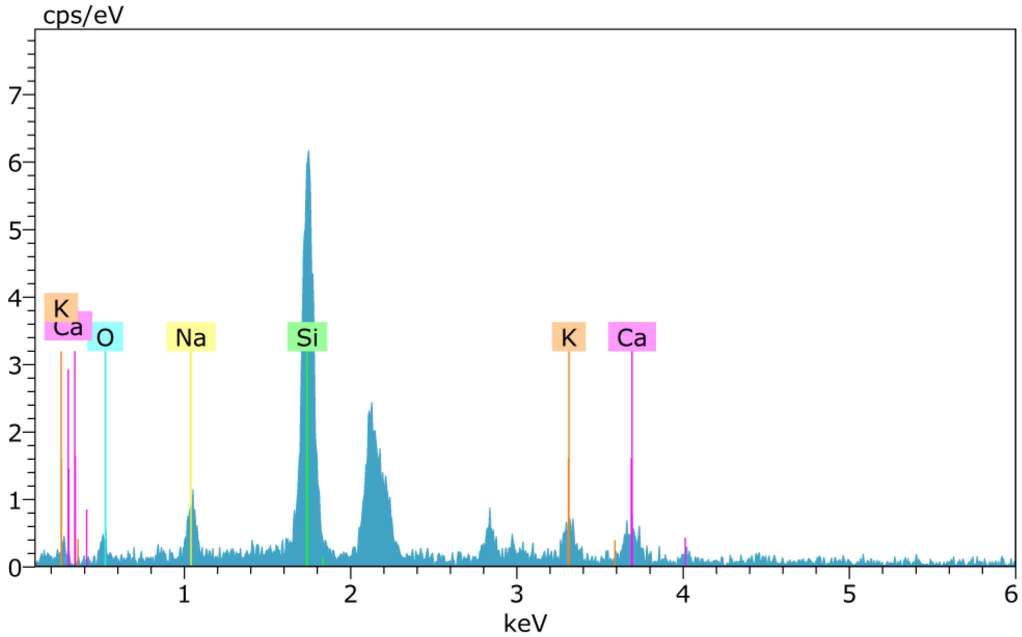

Figure 3. EDS Spectrum of untempered bulk material.

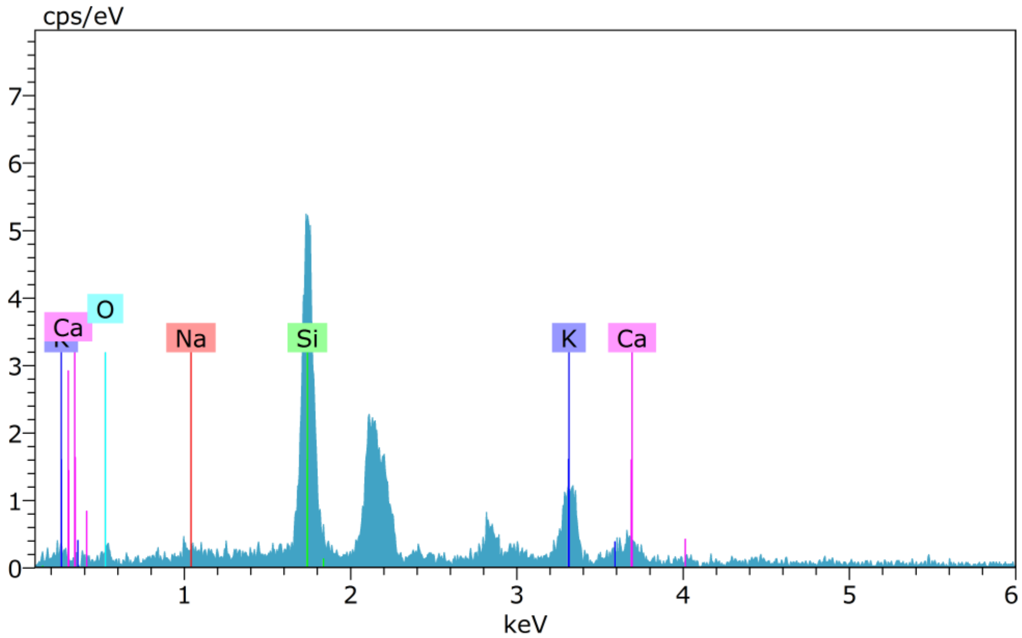

Figure 4. EDS Spectrum of tempered bulk material.

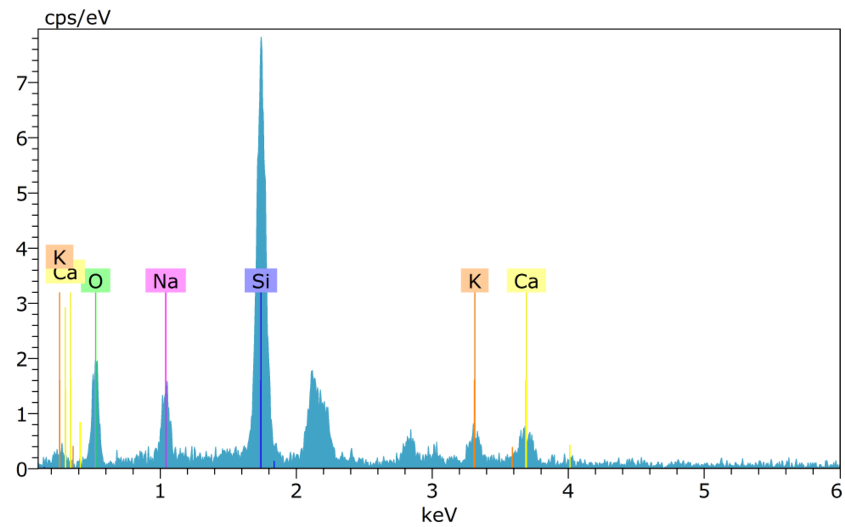

Figure 5. EDS Spectrum of untempered powder material.

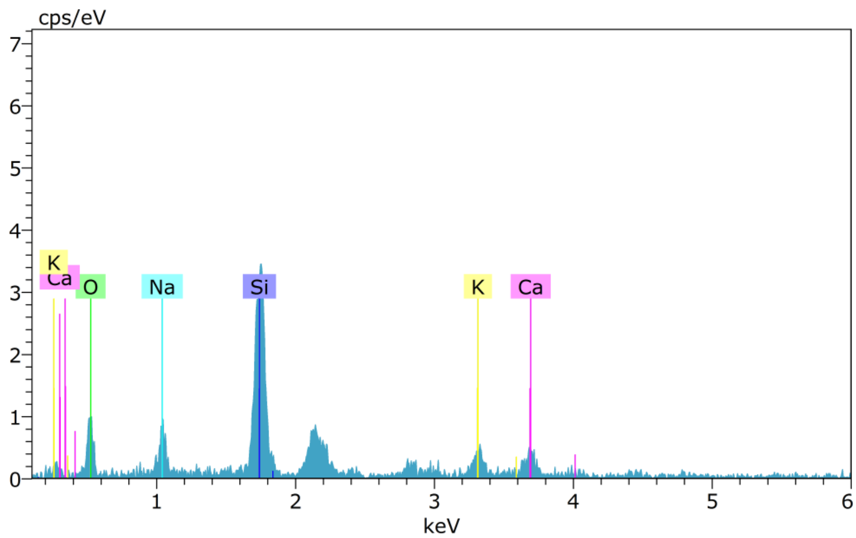

Figure 6. EDS Spectrum of tempered bulk material.

3.3. Hardness analysis

The Vickers hardness measurement method was employed to evaluate the hardness of both untampered and chemically tempered samples. The measurements were conducted using a constant loading rate, a dwell time of 15 seconds, and an applied force of 50 grams during the indentations. The image analysis software of the optical profiler (Bruker Counter GT-K1) was used to determine the hardness values in the measurements of the indentation diagonals Figure 7. An average value based on 10 indentations was calculated to represent the hardness of each sample.

The tests were carried out under controlled environmental conditions, maintaining a temperature of 23 ± 1 °C and a relative humidity of 50-60%. The chemically tempered glass exhibited a hardness of 6.16 GPa ± 0.18, while the untempered glass showed a hardness of 5.53 GPa ± 0.12, as shown in Table 2. These results indicate a significant increase in hardness for the chemically tempered glass compared to the original untempered glass.

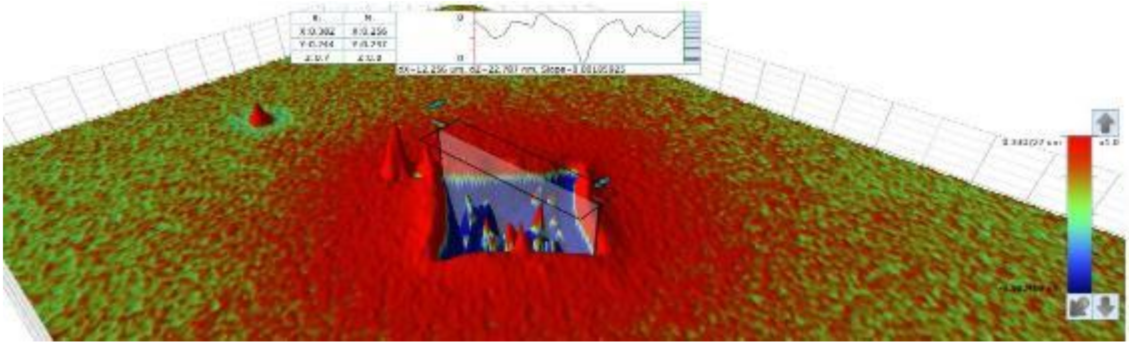

Figure 7. Measurements of recess diagonals using optical profiler.

Table 2. Vickers hardness of untempered original glass sample and chemically tempered glass sample.

Measurement No:	Unprocessed original glass (mm)	Chemical tempered glass (mm)
1	0,00891	0,00844
	0,00897	0,00873
2	0,00909	0,00854
	0,00917	0,00869
3	0,00885	0,00868
	0,00901	0,00864
4	0,00904	0,00871
	0,00922	0,00857
5	0,00915	0,00881
	0,00905	0,00851
6	0,00905	0,0087
	0,00917	0,00851
7	0,00906	0,00879
	0,00908	0,00843
8	0,00918	0,00869
	0,00922	0,0085
9	0,00908	0,00856
	0,00898	0,00844
10	0,009	0,00845
	0,009	0,00843
Average	0,00906	0,00859
HV	564 ± 12	628 ± 18
GPa	5,53 ± 0,12	6,16 ± 0,18

3.4. Mechanical tests

To evaluate sample durability, mechanical tests such as impact resistance, free fall resistance, and bending resistance, were conducted. The impact strength of both chemically tempered and untempered glass samples was measured using the instrument illustrated in Figure 6 and, with impact testing performed on both rim and bowl sections. These tests adhere to DIN 52295 and EN 12980:2000 standards. Additionally, a different tool, as described in Figure 8 and Figure 9, is employed for impact testing on the bottom part of the glass samples. The final results are shown in Table 3, Table 4, and Table 5.

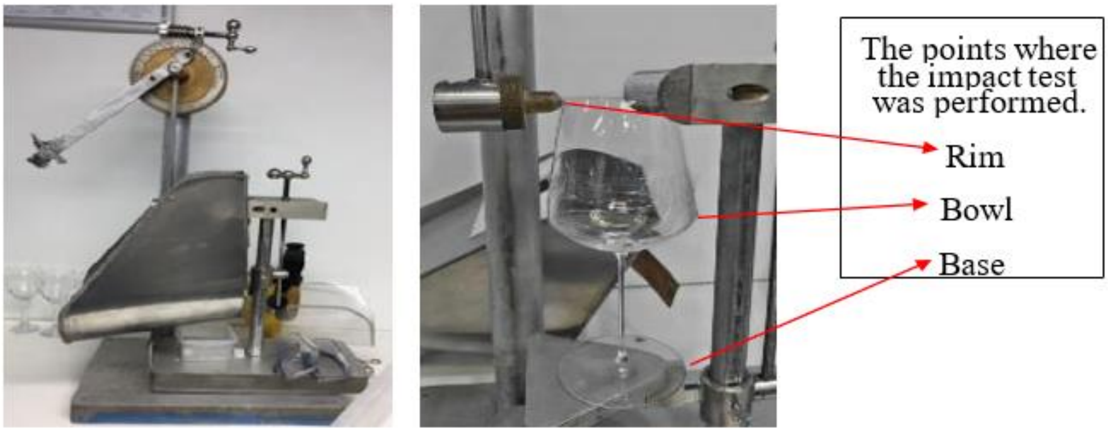

Figure 8. A tool used to test the impact resistance of glass samples taken from the edge and bowl part.

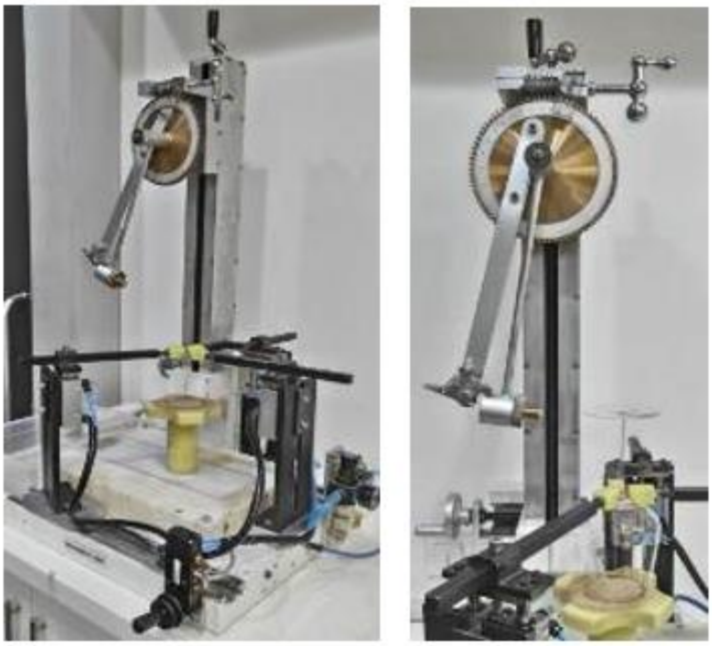

Figure 9. The tool used to test the impact resistance of glass samples taken from the base.

Table 3. Impact test results of the rim

Unprocessed glass			Chemically Tempered glass		
Sample No	**Results (IPS)**	**Thickness of the rim (mm)**	**Sample No**	**Results (IPS)**	**Thickness of the rim (mm)**
R-16-01	30	0,55 – 0,83	C-16-01	45 – 1st	0,62 – 0,71
R-16-02	25	0,83 – 0,88	C-16-02	45 – 3rd	0,58 – 0,59
R-16-03	20	0,50 – 0,56	C-16-03	45 – 1st	0,74 – 0,75
R-16-04	20	0,50 – 0,58	C-16-04	30 – 1st	0,57 – 0,72
R-16-05	25	0,56 – 0,61	C-16-05	45 – 1st	0,62 – 0,93
Average	24	0,64	Average	42	0,68

Table 4. Impact test results of the bowl.

Unprocessed glass		**Chemically Tempered glass**		
Sample No	**Results (IPS)**	**Sample No**	**Results (IPS)**	**Thickness of the bowl (mm)**
R-16-13	15	C-16-06	45 – 2nd	0,52 – 0,60
R-16-14	20	C-16-07	45 – 1st	0,54 – 0,61
R-16-15	20	C-16-08	40 – 1st	0,57 – 0,61
R-16-16	20	C-16-09	40 – 2nd	0,60 – 0,66
R-16-17	20	C-19-10	50 – 1st	0,55 – 0,58
Average	19	Average	44	0,58

Table 5. Impact test results of the base.

Unprocessed glass					
Sample No	**Results (IPS)**	**Break from**	**Thickness of the stem (mm)**	**Thickness of the base (mm)**	**Diameter of the base (mm)**
R-16-06	45	Stem	4,3	2,6	97,7
R-16-07	30	Stem	4,3	2,0	95,5
R-16-08	30	Stem	4,3	2,4	96,8
R-16-09	60	Stem	5,0	2,8	96,6
R-16-10	40	Stem	3,7	2,7	97,5
Average	41		4,3	2,5	96,8
Chemically Tempered glass					
Sample No	**Results (IPS)**	**Break from**	**Thickness of the stem (mm)**	**Thickness of the base (mm)**	**Diameter of the base (mm)**
C-16-11	85	Stem	3,7	2,4	96,8
C-16-12	105	Stem-Base	4,1	2,4	98,0
C-16-13	100	Stem-Base	4,6	2,6	96,5
C-16-14	100	Stem-Base	4,2	2,3	95,7
C-16-15	80	Base	4,8	2,7	96,9
Average	94		4,3	2,5	96,8

Free fall tests were conducted on both chemically tempered and untempered glass samples. Typically, the instrument has a maximum height limit of 50 cm. However, due to the size constraints of the wine glass, it was released for free fall from a height of 28 cm, as shown in Figure 10. The results of the test are provided in Table 6.

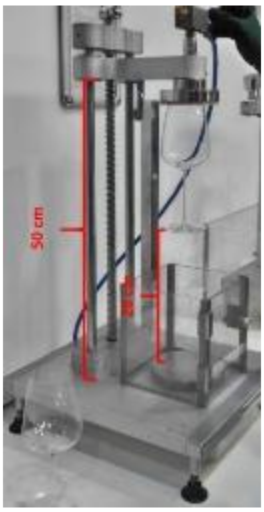

Figure 10. The instrument used for free fall testing.

Table 6. Free Fall test results.

Unprocessed glass		Kimyasal temperlenmiş cam	
Sample No	Results (cm)	Sample No	Results (cm)
R-16-18	18	C-16-16	>28 (no break)
R-16-19	14	C-16-17	>28 (no break)
R-16-20	16	C-16-18	>28 (no break)
R-16-21	14	C-16-19	>28 (no break)
R-16-22	14	C-16-20	28
Ortalama	16	Ortalama	Higher than 28

3.5. Bending test

The bending test was conducted on both chemically tempered and unprocessed glass samples using the test instrument is shown in Figure 9. The results of this test are presented in Table 7. It's worth noting that the maximum bending limit of the instrument is 11 degrees.

Figure 11. Bend Test apparatus.

Table 7. Bending test results.

Unprocessed glass			Chemically tempered glass		
Sample No	Results (degrees)	Thickness of the stem (mm)	Sample No	Results (degrees)	Thickness of the stem (mm)
R-16-23	5,5	4,2	C-16-21	>13 (not break)	3,6
R-16-24	5,5	4,2	C-16-22	>13 (not break)	4,4
R-16-25	6	4,2	C-16-23	13	4,3
R-16-26	6	4,2	C-16-24	>13 (not break)	4,7
R-16-27	6	4,2	C-16-25	>13 (not break))	-
Average	6	4,2	Average	Higher than 13	4,3

3.6. Hydrolytic resistance and dishwasher resistance analysis

Glass samples with Mold number 32016, which are crystalline glass samples, were utilized to detect the changes that may occur in hydrolytic resistance and dishwasher resistance before and after the Chemical Tempering process. ISO 719 "Resistance and classification of glass particles against water at 98°C" and EN 12875 "Resistance of household goods in the dishwasher" tests were carried out. The results obtained are given in Table 8.

Table 8. Water resistance and classification of Glass Particles at 98°C.

Sample Description	mL 0.01 mol/L HCI lg Glass Grains		Hydrolytic Class
	Result (Mean Value)	Limit Value	
32016 Unprocessed Glass	0,72	from 0,20 up to and including 0,85	HGB3
32016 Chemically Tempered Glass	0.55		

Hydrolytic Class	mL 0,01 moVL HCI Glass Grains
HGB 1	up to and including 0,10
HGB 2	from 0,10 up to and including 0,20
HGB 3	from 0,20 up to and including 0,85
HGB 4	from 0,85 up to and including 2,0
HGB 5	from 2,0 up to and including 3,5

It was kept immersed in a 0.5% (w/w) type C detergent (Finish Classic Powder) solution at 75oC. It was examined by the criteria given below according to the standard EN 12875-2. The examination results are given in Table 9.

Table 9. Dishwasher resistance of household items - fast method.

Ranking Degree	Surface Change result
0	No Change
1	First discerniblechange
2	Clearly visible change

Sample Describtion	16 HOURS	32 HOURS	48 HOURS	64 HOURS
Unprocessed Glass	0	0	0	0
Chemically Tempered Glass	0	0	0	0

From the analysis results presented in Table 10, it is evident that the chemically tempered glass exhibits higher resistance to hydrolysis compared to untreated glass. This is indicated by the reduced volume of acid (equivalent amount of sodium oxide) required for the titration of the extracted unit mass of chemically tempered glass. This reduction in required acid signifies lower reactivity with acid and, consequently, higher resistance to the corrosive effects of water.

Regarding dishwasher resistance, no notable difference was observed between the chemically untempered and tempered glass samples with mold number 32016, as per the EN 12875-4 standard. After 64 hours of testing, neither glass sample showed signs of deterioration such as clouding or grain defects. However, it is noteworthy that the untempered glass samples fractured from the foot part during testing, whereas the tempered glass samples remained intact. This indicates that tempered glass has better resistance to breakage under the specified testing conditions.

Table 10. DOL and CS values of the samples which were tested according to EN 12875-4.

Sample Description	Before Test		After 16 H		After 32 H		After 48 H		After 64 H	
	DOL (μm)	CS (MPa)	DOL (μm)	CS (MPa)	DOL (μm)	CS (MPa)	DOL (μm)	CS (MPa)	DOL (μm)	CS (MPa)
Sample 1	19,6 ±2,1	413 ±8	16,0 ±1,7	403 ±10	16,7 ±1,3	393 ±8	15,6 ±2,6	360 ±5	15,5 ±1,1	339 ±10
Sample 2	19,2 ±0,7	425 ±6	17,7 ±2,3	403 ±1	18,1 ±1,2	381 ±3	13,4±2,3	370 ±6	13,9 ±1,2	331

4. Conclusion

In conclusion, this study investigated the effects of mechanical washing resistance and weather conditions on both untempered and chemically tempered glass. The results revealed that chemically tempered glass exhibited higher compressive stress, greater hardness, and superior resistance to breakage compared to untempered glass. Additionally, chemically tempered glass exhibited enhanced resistance to hydrolysis. Overall, tempered glass demonstrated superior breakage resistance under the specified test conditions.

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Conflict of Interest Statement

The authors declare that there is no conflict of interest.

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