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Influences of hardening agent on some physical and mechanical properties of medium-density fiberboard

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Abstract: Effects of Ammonium chloride (NH_4CI) as a hardening agent on thickness swelling (TS), water absorption (WA), screw holding resistance (SHR), Janka hardness, modulus of rupture (MOR), modulus of elasticity (MOE), and internal bonding (IB) properties of medium-density fiberboard (MDF) were evaluated. Target densities were 712 and 715 kg/m³ for hardener applied (0.75 kg/m³ solid as 10% solution (fiber dry wt.)) and unmodified factory made 18 mm thick MDF, respectively. A total of 400 samples were tested. Boards produced without hardener presented better mechanical properties except for SHR. Indeed, SHR was around 9.2% improved by hardener utilization. However, hardener utilization caused around 8.4%, 7.3%, 3.6%, and 1.3% decreases for MOE, MOR, IB, and Janka hardness, respectively. Surprisingly, soaking time caused opposite results for TS and WA. The TS and WA of the hardener utilized MDF decreased around 40.3% and 29.6% for short-term soaking (2h) but remarkable increases (around 62.4% and 20%, respectively) were observed for long-term (24h) soaking. Statistical analysis proved that there were statistically significant ($P<0.05$) differences between all the evaluated properties.
Keywords: Medium density fiberboard, Oak, Hardener, Physical and mechanical properties

Sertleştiricinin orta yoğunluklu lif levhanın bazı fiziksel ve mekanik özelliklerine etkisi

Özet: Sertleştirici olarak Amonyum Klorür (NH_4CI)'ün orta yoğunluklu lif levhanın kalınlığına şişme, su alma, vida tutma direnci, Janka sertlik değeri, eğilme direnci, eğilmede elastikiyet modülü ve çekme direnci özelliklerine etkisi değerlendirilmiştir. Sertleştirici kullanılan (kuru life oranla %10 solüsyon olarak 0.75 kg/m³ katı) ve sertleştirici kullanılmayan (kontrol grubu) fabrika üretimi 18mm orta yoğunluklu lif levhaların hedef yoğunlukları, sırası ile 712 ve 715 kg/m³'tür. Toplam olarak 400 örnek test edilmiştir. Sertleştirici kullanılmayan levhalar, vida tutma direnci hariç, daha iyi mekanik özellikler sergilemiştir. Sertleştirici kullanımı ile vida tutma direnci yaklaşık %9.2 iyileştirilmiştir. Fakat sertleştirici kullanımı, elastikiyet modülü, eğilme direnci, çekme direnci ve Janka sertlik değerlerinde sırası ile yaklaşık %8.4, %7.3, %3.6 ve %1.3 düşüşe neden olmuştur. Şaşırtıcı şekilde, suya daldırma süresi kalınlığına şişme ve su almada zıt sonuçlara sebep olmuştur. Kısa süreli suya daldırmada (2 saat) sertleştirici kullanılan levhaların kalınlığına şişme ve su alma değerleri sırasıyla yaklaşık %40.3 ve %29.6 azalmış iken uzun süreli (24 saat) suya daldırmada kayda değer (sırasıyla yaklaşık %62.4 ve %20) artışlar gözlenmiştir. İstatistiksel analizler, değerlendirilen tüm özelliklerde istatistiksel olarak anlamlı ($P<0.05$) farklılıklar olduğunu ortaya koymuştur.
Anahtar kelimeler: Orta yoğunluklu lif levha, Meşe, Sertleştirici, Fiziksel ve mekanik özellikler

1. Introduction

Wood-based composites are some of the essential engineered products used for construction and building purposes and medium-density fiberboard (MDF) is one of the most commonly used board types. The MDF is commonly used as an alternative for solid wood, plywood, and particleboard (PB) in lots of furniture practices. Furthermore, interior door skins, mouldings, and interior trim parts are other utilization types of MDF (Stark et al. 2010).

Fiber, glue, and wax (Uner and Olgun 2017) are the common components of the MDF. Typically used adhesive for primarily PB and MDF for interior applications are Urea-formaldehyde (UF) resins due to their some advantages such as lower curing temperatures than phenol-formaldehyde (PF) resins and application ability at different curing circumstances while formaldehyde release from the boards bonded with UF is a health interest (Stark et al. 2010). To reduce such disadvantages and improve physical and mechanical properties, lots of modification agents have been utilized in MDF such as maleic anhydride (Hundhausen et al. 2015), nano-boron nitride (BN), and nano-titanium dioxide (TiO_2) (Kızılkaya et al. 2020), zeolite (Çamlıbel and Yılmaz Aydın 2020), rock salt (Çamlıbel and Akgül 2020), borax pentahydrate (Akgül and Çamlıbel 2021), activated carbon (Akın and Karaboyacı 2021), activated charcoal (Darmawan et al. 2010), etc. However, hardener type is one of the critical determinants for the properties of boards and has significant influences on the board characteristics (Atar et al. 2014), and ammonium sulfate ($(NH_4)_2SO_4$) and ammonium chloride (NH_4Cl) are

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the most common hardeners utilized to arrange pH and cure the resins (Uner and Olgun 2017). When the literature is reviewed, the followings are some of the studies that evaluated hardener utilization in the wood-based composite.

Bekhta et al. (2016) evaluated the influence of hardener type (aluminum chloride $AlCl_3$, aluminum sulfate $Al_2(SO_4)_3$, ammonium persulphate $(NH_4)_2S_2O_8$, Ferrum chloride $FeCl_3$, and combined hardener) on some properties of plywood. Aras et al. (2015) evaluated the influence of hardener type (NH_4Cl and ammonium nitrate NH_4NO_3) on the density, thickness swelling (TS), water absorption (WA), modulus of rupture (MOR), modulus of elasticity (MOE), internal bond strength (IB), screw holding resistance (SHR), surface durability and formaldehyde content of PB. Çamlibel (2020) produced 18 mm thick PB using 20% NH_4Cl (0.95g/cm^3 density) as a hardener and a 40:30:20 mixture of *Pinus slyvestris* L., *Quercus petraea* L. and *Populus alba* L. and 10% sawdust. İstek and Özlüsoylu (2021) used 1% NH_4Cl for the production of fiberboard using 45:55 of red pine and beech species, respectively, and evaluated the influence of pressing temperature and duration on TS, WA, MOE, MOR, and SHR properties. Önem and Kaymakçı (2019) produced 18 mm thick MDF using 10% NH_4Cl and beech fiber and evaluated the pressing parameters on the flatness of single face-coated MDF. Ayrılmış et al. (2011) used 1% of NH_4Cl solution based on the resin solid content (20%) as a hardener in UF resin for MDF production with rubberwood fiber and evaluated the influence of press parameters (temperature and duration) on density, TS, WA, MOR, MOE, IB, and SHR. Ayrılmış et al. (2010) used *R. Ponticum* fiber, 1% paraffin, 1% NH_4Cl (30% solid content), and 11% UF resin for the production of 18mm thick MDF, and evaluated surface properties. Akgül et al. (2013) evaluated the usability of Luffa fiber on MDF production with 0.8% NH_4Cl, 11% UF resin, and 1% paraffin. Moreno-Anguiano et al. (2022) produced 12mm thick MDF using *A. durangensis* bagasse and wood fibers (0:100, 10:90, and 30:70, respectively), 1% wax, 14% UF resin, NH_4Cl (25%), and evaluated the TS, WA, MOE, and MOR. Bono et al. (2006) applied 1% NH_4Cl for hardening melamine urea-formaldehyde (MUF) resin and evaluated curing behavior. Lu et al. (2021) produced UF resin bonded MDF using NH_4Cl (1% to solid resin content) as a hardener. Atar et al. (2014) reported that particles with NH_4Cl present lower pH value than $Al_2(SO_4)_3$ and hardener decreased pH values of wood particles with UF resin due to acid catalyzing behavior for the curing reaction of resin.

Different additives and production parameters have been evaluated to improve board properties but as Halvarsson et al. (2008) expressed; adhesion degree or ability between the main components is one of the major determinants for the mechanical properties of the boards. These determinants are dependent on the right formulation of the composition. Even if the companies generally specify systematically to obtain the right mix ratios of resin and hardener which can be easily prepared by mete out by weight or volume (Park and Seo 2011), evaluating the influences of different ratios and modification agent utilization is the essential either for scientific community or business. Therefore, the objective of this study is to figure out the influence of NH_4Cl on some physical and mechanical properties of MDF.

2. Materials and methods

The 18x1830x3660 mm sized panels were produced using Oak (*Quercus robur)* fibers with hardener (WH) and without hardener (WoH). Woods were purchased from Bolu Forestry Department, Turkey. Ammonium chloride (NH_4CI) was used as a hardening chemical. The target densities of the WH and WoH panels were 712 kg/m³ and 715 kg/m³, respectively. Except for hardener utilization, production parameters were constant and are presented in Table 1.

Panels were produced using active production lines in a board production facility instead of laboratory-type machines or tools. Oak logs were chipped and stored in bins. Chips were screened using a mechanic oscillating screener. Obtained standard chips with 3.9 pH were pre-steamed at 135°C temperature and 2.6 bar steam pressure using a refiner. Chips were fed to Asplund-defibrator (digester) using a plug screw. They were cooked at 183°C temperature and 7.5 bar steam pressure for 3.5 minutes and then, fibers were produced using cooked chips were fed to a refiner for defibrillation processes by discharge screw. The pH of the fiber was 4.5. Liquid paraffin (1.15 kg/m³, wt% dry fiber) was applied to the discharge screw. NH_4CI (6.5 pH, as 10% solution and 0.75 kg/m³ (fiber dry wt.)) was blended with fiber in the blow line. The UF resin (7.5-8.5 pH, 65% solid matter and 12% dry fiber wt.) was applied (pulverized) to fibers in the blow line. Fibers were dried at around 12% moisture content. Homogeneously blended fibers were transferred to the mat forming station and 110-90-105 kg/cm² pressures were applied in the pre-pressing application for the mat formation. Mats were edge-trimmed (1860x3690mm) and loaded to multiday press. At a time 8 mats were hot-pressed using parameters seen in Table 1. Produced MDFs were cooled using star type cooler and then sized. Boards were stacked 5 days in storage. The surfaces of the boards were sequentially sanded using 40, 80, and 120 sandpapers to achieve 18mm thickness.

Boards were acclimatized at 20±2°C temperature and 65±5 relative humidity to reach around 12% moisture content in accordance with TS 642-ISO 554 (1997). A total of 400 samples were prepared and density (TS EN 323 1999), WA for 2 and 24 h (TS EN 317 1999), TS for 2 and 24 h (TS EN 317 1999), MOE in bending (TS EN 310 1999), MOR (TS EN 310 1999), IB (TS EN 319 1999), SHR (BS EN 320 2011), and Janka hardness (ASTM D-1037-78 1994) were figured out according to cited standards.

Table 1. MDF production parameters

Board type	Fiber (Oak)	Adhesive	Paraffin	Hardener (NH_4Cl)	Multiday pressing				Size
	%	UF (1.17 moles) Solid (kg/m³)	Liquid (kg/m³)	Solid (kg/m³)	Time (s)	Pressure (Kp/cm²)	Temperature (°C)	Speed (mm/s)	(mm)
WH WoH	100	83	1.15	0.75 -	275	32	185	145	18x1830x3660

3. Results and discussion

Average values and statistics for the physical and mechanical properties of MDFs are presented and illustrated in Table 2 and Figure 1, respectively. According to the results, there are scarcely any (0.53%) differences between the densities. Therefore, the influence of density discrepancies on the physical and mechanical properties may be disregarded. As can be seen in the table, outstanding properties that were oppositely influenced by hardener utilization are TS and WA in terms of soaking time. Hardener utilization provided around 40.3% and 29.6% improvement for 2h applications of TS and WA, respectively. However, 24 h soaking caused around 62.4% and 20% worsening in swelling and absorption behavior. According to TS EN 312 (2012), the upper bounds of swelling in thickness percentages range from 10% (Types P5 and P7; structural and heavy-duty structural purposes in humid conditions, respectively) to 15% (Types P4 and P6; structural and heavy-duty structural purposes at dry conditions, respectively) for boards with a thickness higher than 13mm and up to 20mm. Furthermore, 14 is the maximum allowable TS percentage for Type P3 (non-structural purpose in humid conditions) boards. Therefore, as can be seen in Table 2, except for P5 and P7 requirements (due to a 5.6% difference), WoH boards meet the standard. On the other hand, boards produced using hardener did not meet the requirements for all the board types mentioned in the standard. Therefore, it should be noted that boards produced without hardener may present better performance for prolonged contact with the humid environment. Due to its hydrophilic nature, wood particles absorb water molecules that cause dimensional changes by water immersion. As a result of dimensional increases, mechanical properties would decrease (Wang and Tsang 2018).

Except for SHR, all the mechanical properties were decreased when boards were produced using a hardener. The highest decrease (8.4%) was observed for MOE while the lowest (1.3%) was for Janka hardness. On the contrary, SHR was the highest (9.24%) influenced property by hardener utilization but in a positive manner.

The IB may be a sign of the penetration depth of resin into the internal structure and if the penetration is not well enough, IB at the hearth will not be good enough which will be the reason for the delamination or fall apart of the surfaces (Hutten 2007). According to TS EN 312 (2012) standard, IB ranges from 0.24MPa (Type P1; general-purpose at dry conditions) to 0.7MPa (Type P7). Furthermore, 0.35MPa, 0.45MPa, and 0.5MPa are the minimum requirements for P2 (interior purposes including furniture under dry conditions) and P4, P3 and P5, and P6 type boards, respectively. When compared to standard, it's seen that only the P7 requirement could not be met by the produced boards. Therefore it can be said that penetration of resin was fair enough. However, 0.6 and 0.9 MPa values were reported by Levy (2012) for interior and exterior utilization of MDF (640-800 kgm^3) which has up to 21mm nominal thickness but these values were noteworthy higher than the standard values.

Strong correlations (R^2: 0.61, 0.74, and 0.83) between SHR and IB for PB were reported by Semple and Smith (2006). However, opposing behavior was seen between two variables by hardener utilization and should be evaluated by a future study. Because as Tor et al. (2016) reported, the structural unity of a product is directly correlated to the connection ability or performance of the members. And, SHR is one of the performance indicators that is adversely affected by soaking to water (Yorur et al. 2020).

As can be seen in the table, the stiffness of MDFs was adversely affected by hardener utilization. However, when the requirements of TS EN 312 (2012) standard are taken into consideration, the strength and elasticity properties of all the boards are higher than the required values. Furthermore, these values are higher than those Levy (2012) reported for interior purposes. Therefore, either unmodified or hardener modified boards are suitable for the commercial market.

Hardener kind, concentration, and proportion of hardener and resin are some of the significant determinants to achieve appropriate stiffness (Uner and Olgun 2017). According to Atar et al. (2014) board produced using NH_4Cl provided higher flexural properties than those of ammonium sulfate $(NH_4)_2SO_4$, and authors reported that the best board characteristics (MOR, MOE, IB, TS, formaldehyde emission, surface roughness, and contact angle) were achieved with NH_4Cl, followed by $(NH_4)_2SO_4$ and $Al_2(SO_4)_3$. According to the results of this study, an entire improvement for all the board characteristics was not obtained by using NH_4Cl. Indeed, the opposite behavior is obvious, particularly for TS and WA.

Ayrılmış (2002) produced 18 mm thick MDF using *Quercus robur* L. fiber, 1% wax, 0.8% NH_4Cl as a hardener, and 11% UF resin and evaluated the density, TS, and WA for 2 and 24h properties. Reported values for density, TS 2 and 24h, and WA 2 and 24h were 0.758g/cm^3, 1.88%, 7.40%, 4.3% and 17.03%, respectively. These values were around 6.46% higher, and 17.5%, 56.9%, 71.3%, and 65.9% lower than the results of this study. These significant numerical differences may be occurred due to pressing parameters and compositing of the mat.

Table 2. Averages and group statistics for physical and mechanical properties

	Property	Panels	N	Mean (%)*	Std. Dev.	Std. Error
Physical	Density (kg/m³)	WoH	20	715.78	3.54	0.79
		WH	20	712.02 (-0.53)	3.56	0.80
	TS 2h (%)	WoH	20	3.82	0.40	0.09
		WH	20	2.28 (-40.31)	0.78	0.18
	TS 24h (%)	WoH	20	10.56	0.29	0.06
		WH	20	17.15 (+62.41)	0.54	0.12
	WA 2h (%)	WoH	20	21.30	2.01	0.45
		WH	20	15.00 (-29.58)	1.12	0.25
	WA 24h (%)	WoH	20	41.68	2.87	0.64
		WH	20	50.00 (+19.96)	0.84	0.19
Mechanical	SHR (MPa)	WoH	20	10.07	0.30	0.07
		WH	20	11.00 (+9.24)	0.48	0.11
	Janka Hardness	WoH	20	81.05	1.23	0.28
		WH	20	80.00 (-1.30)	0.73	0.16
	MOR (MPa)	WoH	20	36.89	2.44	0.54
		WH	20	34.21 (-7.27)	0.61	0.14
	MOE (MPa)	WoH	20	3482.92	218.22	48.79
		WH	20	3191.36 (-8.37)	85.97	19.22
	IB (MPa)	WoH	20	0.59	0.03	0.01
		WH	20	0.57 (-3.58)	0.01	0.00

N sample size, * % difference from averages of the unmodified boards, WH with hardener, and WoH without hardener.

Relationships between two independent group averages are presented in Table 3. As can be seen in the table, equal variances (P>0.05) were assumed for density, WA 2h, SHR, and Janka hardness while others were not. However, according to Sig. (2-tailed) values, there are significant differences (P<0.05) between the means of WoH and WH for all the evaluated properties. Statistically significant reductions in the mechanical properties were seen but it should be noted that boards produced using NH_4Cl as a hardener agent can provide better joints for furniture production using screws.

Curing duration is related to some parameters such as temperature, resin type, modification chemicals, etc. Duration of curing is a period required for the curing of the resin in the existence of hardener (Bono et al. 2006) and this period can be arranged by set-up the temperature and hardener utilization. Curing reactions of UF can be accelerated by increasing the temperature and decreasing the curing period has significant influences on productivity and productions costs (Uner and Olgun 2017). However, these parameters should be optimized so as not to reduce the physical and mechanical properties of the boards as in this study.

Wu et al. (2006) evaluated the influence of hardener composition on the curing and aging of epoxy resin. The authors stated that the curing rate increases with the amount of hardener, and moisture can incredibly quicken post-cure, and WA increases with an increase in hardener amount.

The UF resin needs acidic circumstances and pH is generally arranged at about 4 for curing. For that reason, hardeners are required for proper curing of the UF either applied at room or higher temperatures. Furthermore, NH_4CI is one of the most commonly used hardener for controlling the pH and curing the adhesive (Uner and Olgun 2017), and NH_4CI with 6.5 pH was used for control and cure purposes in this study.

Table 3. Statistics for independent samples test

	Levene's test for equality of variances			t-test for equality of means					
Property	Equal variances	F	Sig.	t	df	Sig. (2-tailed)	Mean difference	Std. error difference	95% CI of the difference Lower	Upper
Density	A*	0.398	0.532	3.349	38	0.002	3.76	1.12	1.49	6.03
(kg/m³)	NA**			3.349	37.999	0.002	3.76	1.12	1.49	6.03
MOR	A	41.358	0.000	4.773	38	0.000	2.68	0.56	1.54	3.82
(MPa)	NA			4.773	21.390	0.000	2.68	0.56	1.51	3.85
MOE	A	8.793	0.005	5.559	38	0.000	291.56	52.44	185.39	397.73
(MPa)	NA			5.559	24.759	0.000	291.56	52.44	183.49	399.62
IB	A	26.962	0.000	2.552	38	0.015	0.02	0.01	0.00	0.04
(MPa)	NA			2.552	22.804	0.018	0.02	0.01	0.00	0.04
TS 2h	A	4.722	0.036	7.805	38	0.000	1.54	0.20	1.14	1.93
(%)	NA			7.805	28.198	0.000	1.54	0.20	1.13	1.94
TS 24h	A	6.012	0.019	-47.986	38	0.000	-6.59	0.14	-6.87	-6.32
(%)	NA			-47.986	28.883	0.000	-6.59	0.14	-6.88	-6.31
WA 2h	A	3.462	0.071	12.237	38	0.000	6.30	0.51	5.26	7.34
(%)	NA			12.237	29.838	0.000	6.30	0.51	5.25	7.35
WA 24h	A	16.802	0.000	-12.439	38	0.000	-8.32	0.67	-9.67	-6.96
(%)	NA			-12.439	22.205	0.000	-8.32	0.67	-9.70	-6.93
SHR	A	1.679	0.203	-7.368	38	0.000	-0.93	0.13	-1.19	-0.67
(MPa)	NA			-7.368	32.169	0.000	-0.93	0.13	-1.19	-0.67
Janka	A	2.765	0.105	3.280	38	0.002	1.05	0.32	0.40	1.70
Hardness	NA			3.280	30.727	0.003	1.05	0.32	0.40	1.70

*assumed, ** not assumed

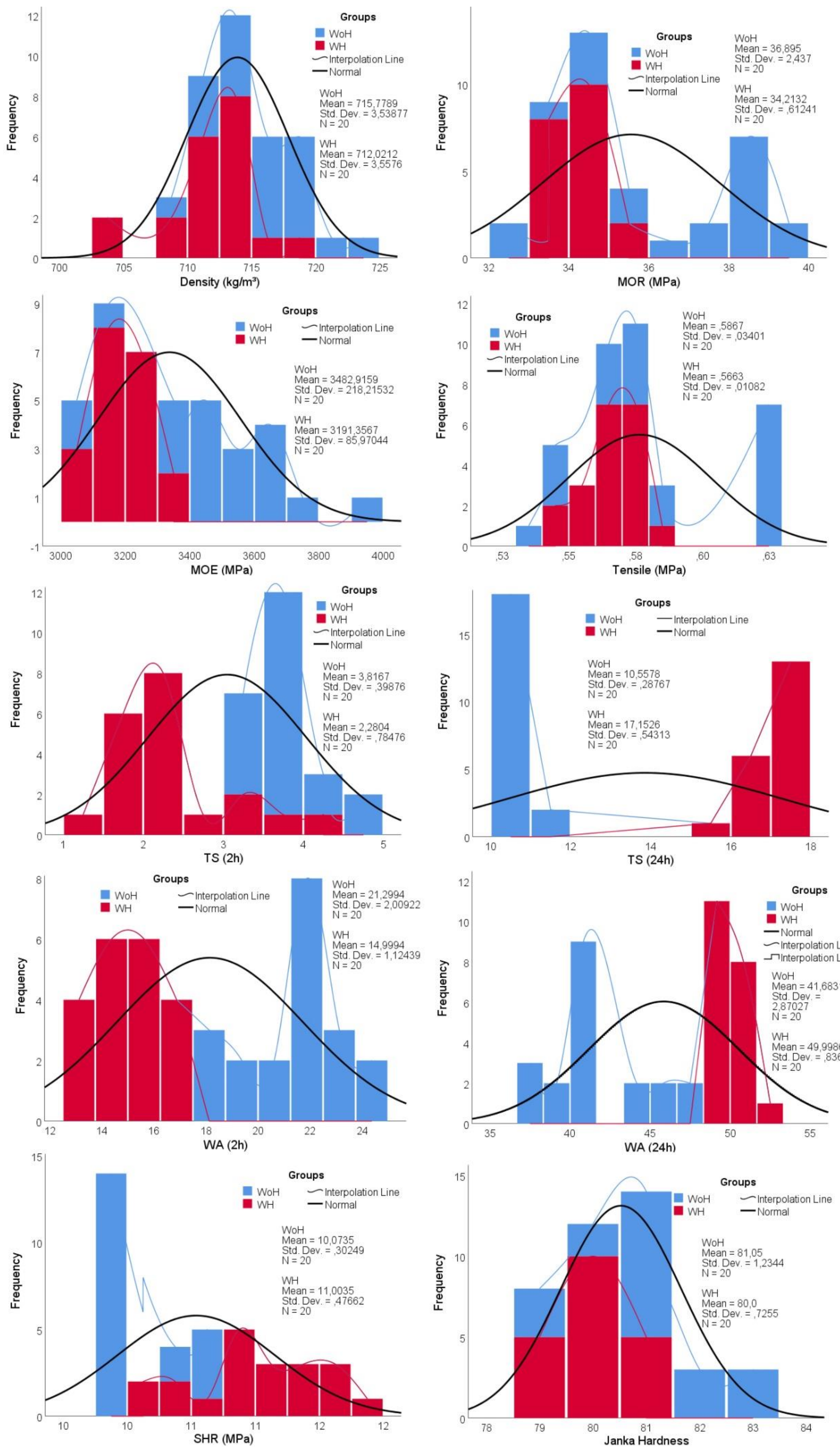

Figure 1. Histograms with interpolation and distribution curves of the properties in terms of WoH and WH groups.

4. Conclusion

Materials (fiber, resin, modification agents such as paraffin, hardener, etc.) and production parameters such as pressing (time, pressure, temperature, speed, etc.) are the determinants of board characteristics. In this study, the influences of Ammonium chloride utilization as a hardener on some physical and mechanical properties were evaluated.

Ammonium chloride blended UF resin bonded MDF presented remarkable decreases in flexural strength and deflection ability. Following the first two (MOE and MOR), IB and Janka hardness were the third and fourth adversely influenced mechanical properties. On the other hand, screw holding ability was considerably improved by hardener utilization.

Even decreases were seen for mechanical properties by hardener utilization, both WoH and WH boards have met the requirements of EN 312 standard.

Hardener utilization provided significant improvement on WA and TS of the boards for short-term soaking while long-term soaking caused opposite behavior. It can be said that boards produced without hardener may be more suitable for long-term utilization in humid conditions due to higher resistance to WA and swelling.

Besides physical and mechanical properties, hardener influences the curing time of the resin which is directly related to production volume hence the cost and market share. Therefore, a comprehensive study should be performed to figure out not only the board properties but also business parameters.

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