

The Effects of Different Technological Treatments on *Ficus retusa* Branchwood Particleboard: Physical Properties and Formaldehyde Emission Evaluations

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ABSTRACT

This research aimed to compare the effect of post-manufacture thermal treatment and wax emulsion (1%) as an additive on water absorption, thickness swelling, and formaldehyde emission of particleboard produced from *Ficus retusa* branchwood. The particleboards were single layer produced with a target density of 690 kg. m⁻³ and 12% urea formaldehyde as a binder. Three different temperatures (180, 200, and 220 °C) and two different heat exposure durations (5 and 10 minutes) were applied. The statistical tests revealed that the post-manufacture heat treatments of the *Ficus* particleboard at 200 °C/10 min and 230 °C/5 and 10 min were effective in improving the dimensional stability (water absorption and thickness swelling) of the panels and reducing the formaldehyde emission. Additionally, using the 1% wax emulsion in the panels enhanced the dimensional stability and was comparable with the 220°C/10 min treatment, while it had no significant effect on the formaldehyde emission. Moreover, the *F. retusa* branchwood was suitable for particleboard production using the production parameters introduced in this study.

Keywords: Particleboard, formaldehyde release, desiccator method, post-manufacture heat treatment, wax emulsion.

INTRODUCTION

Wood composites are one of the most important elements found in homes, offices, and many other indoor places. Wood composites have spread widely due to the great development in their production and the diversity of raw materials. Besides, the rapid technological development of wood composites, adhesives had a major impact on the development of this industry. Wood based-panels can be manufactured in any size and the technological properties of the resulting panels can be controlled (Chapman, 2006).

Various wood composites such as veneer laminated plywood, blockboard, particleboard, medium-density fiberboard, and several other wood products are widely utilized in Egypt. Egypt has limited forest resources for lumber production; however, several factories produce

wood composites relying on some fast-growing trees, bagasse, rice straw, flax shives, and imported poplar, oak, and beech woods for decorative veneer (Hassan *et al.*, 2020). Besides, imported pine and spruce for blockboard production (Hassan, 2019). The demand for such products is increasing dramatically (Hassan *et al.*, 2020). Pruning waste from trees has become an important issue due to unmanaged use and improper disposal of this waste in some countries which may negatively affect the environment. In Egypt, each year due to the pruning as a silvicultural process of *Ficus retusa* which produces a lot of waste woody material, part of this resource is usually used in charcoal manufacturing. To obtain technological utilization of that huge amount of *F. retusa* waste generated from the pruning process per year, there is a need to find new industrial value-added products for this lignocellulosic raw material.

Generally, dimensional stability (swelling in thickness and water absorption) and mechanical behavior are essential characteristics to be determined for particleboard quality assessment. Moreover, formaldehyde release from particleboards is also an important test as it adversely affects human health (Lee *et al.*, 2017). Despite this technological development in this industry, a major environmental problem arose from it because of the emission of harmful substances to human health called volatile organic compounds (VOCs), from them, is formaldehyde (Kim and Kim, 2005). Formaldehyde has serious effects on human health, as it is now classified as a carcinogenic substance and affects negatively on the respiratory system and causes eye irritation (Pizzi, 1994; Norbäck, 2009; Song *et al.*, 2015). Generally, the main reason for the formaldehyde liberation from wood based-panels is the presence of adhesives containing formaldehyde (Song *et al.*, 2015). The most common of such adhesives are urea formaldehyde and phenol formaldehyde; both adhesives are widely used due to its low cost and good performance (Pizzi, 1994). For example, in the urea formaldehyde adhesives, the degradation of methylol

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groups and the unreacted formaldehyde cause formaldehyde liberation from in-service wood products (Marutzky, 1994; Dunky, 1998; Hemmilä *et al.*, 2018). It is worth noting that the wood itself can emit formaldehyde (Birkeland *et al.*, 2010).

There are various procedures are now used for formaldehyde liberation determination from wood and wood composites, from these procedures, the environmental chamber that adopted by the American, European, and Japanese standards, besides, the dynamic micro-chamber method that is approved in North America, and another method that is based on the board formaldehyde content through extraction called perforator. In addition to the previous methods, two simple methods depend on the emitted formaldehyde from wood products under specified conditions; they are the flask and desiccator. The desiccator test methods are extensively used by several standards and adopted to be used as a quality control method in Japan, Europe, and the USA. The desiccator (JIS A 1460, 2001) standard is a widely utilized test in Asia and internationally (Que and Furuno, 2007; Risholm-Sundman *et al.*, 2007; Hemmilä *et al.*, 2018). Of course, there are various technologies are now used to reduce the formaldehyde release from wood products. From them, resin modification through reducing the molar ratio of formaldehyde to urea in the urea formaldehyde adhesives, however, lowering the molar ratio under certain limit has a negative effect on the physical and mechanical properties of the produced panels (Que *et al.*, 2007). In a study by (Hassan, 2009) who produced particleboards from young Eucalypt wood using urea formaldehyde with two different molar ratios, the results indicated that the low molar ratio resin reduced the formaldehyde liberation from the panels and had acceptable physical and mechanical properties. As the previous method may affect the bond quality, great attention has been directed to resin additives, such as formaldehyde catchers (Pizzi, 1994; Kim and Kim, 2005; Uchiyama *et al.*, 2007). Other methods could be applied after panel manufacturing reviewed by (Myers, 1986) such as exposing the particleboard to ammonia gas and the applying of surface coatings. Moreover, board-covering methods such as decorative paper overlay affected positively on reducing the formaldehyde emission (Groah *et al.*, 1984).

Several methods are used to modify wood properties; from these methods is heat treatment (Hill, 2006). The thermal treatment is commonly used to improve solid wood properties such as decay resistance and dimensional stability (Esteves, 2009). Some studies have applied the thermal treatment to wood composites after manufacture. Most of these studies focused on

MDF and OSB, and few studies performed on the urea formaldehyde-bonded particleboard (Okino *et al.*, 2007; Lee *et al.*, 2017; Ayrlmis and Winandy, 2009; Pan *et al.*, 2010).

The effect of this post-manufacture thermal treatment on formaldehyde emission from wood products is not well studied in the literature. Only a study found in the literature by (Ates *et al.*, 2017) applied the thermal treatment to MDF panels aiming to reduce their formaldehyde emission levels. Therefore, in response to this gap in the literature, there is a need to explore the effect of various temperatures and exposure durations, especially for particleboard. Additionally, the post-manufacture thermal treatment of particleboard produced from *F. retusa* branchwood is still practically unexamined. Therefore, the objective of this research was to compare the effect of post-manufacture heat treatment and wax emulsion (1%) as an additive on the dimensional stability and formaldehyde emission of particleboard produced from *Ficus retusa* branchwood.

1. Particleboard manufacture

One-layer particleboards were prepared from *Ficus retusa* branchwood particles without bark (40-60 mesh). The branches as a raw material were collected from Alexandria, Egypt. The diameter of the branches ranged from 15 to 25 cm. The chemical composition of the raw material was determined to fully characterize this raw material. Total extractive content was determined according to ASTM D 1105-96 (2013), ash content following ASTM D 1102-84 (2013), lignin according to ASTM D 1106-96 (2013), and Kürschner cellulose content was determined according to (Browning, 1967). Additionally, the fiber length of the branchwood was determined according to Franklin's method (Franklin, 1946). 12 % commercial urea formaldehyde (UF) resin was used as a binding agent for the particleboard manufacturing based on the oven-dried weight of the particles. The UF specifications are presented in Table 1.

Table 1. Urea formaldehyde adhesive specifications.

Solid content	55%
Viscosity	300 cps
Density	1.2 g.cm ⁻³
pH	8.3

1 % NH₄Cl (25%) was added as a hardener based on the oven-dried weight of the particles. The dimensions of the panels were 30 x 30 x 0.9 cm with a target density of 0.69 g.cm⁻³. Pressing time, temperature, and pressure were 6 min, 160°C, and 2.5 MPa, respectively. The produced particleboards were kept in a climatic chamber

at 65% relative humidity and temperature of 23 °C for three weeks.

2. Post-manufacture heat treatment of the produced particleboards.

The samples prepared for water absorption, thickness swelling, and formaldehyde emission testing were subjected to heat using a hydraulic hot press with a very small pressure only to ensure good contact between the hot press platens and the panel's surface. Three temperatures (180, 200, and 220°C) and two exposure durations (5 and 10 minutes) were used. The samples were then conditioned at 65% relative humidity and 23 °C temperature until it reached the equilibrium moisture content to be ready for physical and formaldehyde emission testing.

3. Particleboard preparation with wax emulsion.

The panels were produced using the same manufacturing parameters as previously mentioned except that in these panels, 1% wax emulsion (50% solids) was added to the furnish based on the oven-dried weight of particles. The wax emulsion was added after applying the resin.

4. Board evaluation

4.1. Thickness swelling and water absorption determination.

Thickness swelling (TS) and water absorption (WA) after soaking in water for 2 h and 24 h were determined for all the board types on samples with a dimension of 50 x 50 x 9 mm according to EN 317 (1993).

4.2. Formaldehyde emission testing

Pre-conditioned samples for formaldehyde emission testing were prepared following the Japanese Industrial Standard (JIS A 1460, 2001) without edge sealing. The duration of this test is 24 hours under controlled conditions of temperature and relative humidity. The emitted formaldehyde was collected by distilled water placed in a vessel at the desiccator bottom. The dissolved formaldehyde in the distilled water was determined according to (Nash 1953). 2,4-pentanedione and ammonia were added to the formaldehyde solution to produce a yellow solution of 3,5-diacetyl-1,4-dihydrolutidine, then measured using Laxco UV-VIS spectrophotometer (Alpha 1502, Laxco Inc., USA) at a wavelength of 412 nm. Formaldehyde standard solution (Aldrich, formaldehyde, 37-wt % solution in water) was used for performing a calibration curve.

5. Statistical analysis

The analysis of variance (ANOVA) followed by Fisher's tests were used to detect the differences in the properties of the untreated, 1% wax emulsion, and the thermally treated panels at 0.05 level of significance.

Moreover, factorial analysis of variance (3 x 2) was used to investigate the effect of heating temperature, heat exposure time, and their interaction on the tested physical properties and formaldehyde emission of the heat-treated panels at 0.05 significance level.

RESULTS AND DISCUSSION

The chemical constituents were determined in this study and the mean values are presented in Table 2. The α -cellulose, lignin, total extractives, and ash content mean values were found to be 47.76, 29, 6.76, and 2.19 %, respectively. Generally, the ranges of cellulose and lignin of temperate hardwoods are 38-49% and 23-30%, respectively (Rowell *et al.*, 2005).

The mean value of fiber length was found to be 0.8 mm (Figure 1). Ogunkunle and Oladele, (2008) found that stemwood fiber length of 12 *Ficus* species ranged from 1-1.3 mm. The chemical composition and fiber length values of *F. retusa* branchwood were close to several hardwood species that are suitable for particleboard manufacturing.

The mean values for thickness swelling after 2 h (TS_{2h}) and 24 h (TS_{24h}) of water soaking and water absorption after 2h (WA_{2h}) and 24h (WA_{24h}) of water soaking for the heat-treated, 1% wax emulsion, and untreated *Ficus* particleboard are shown in Table 3.

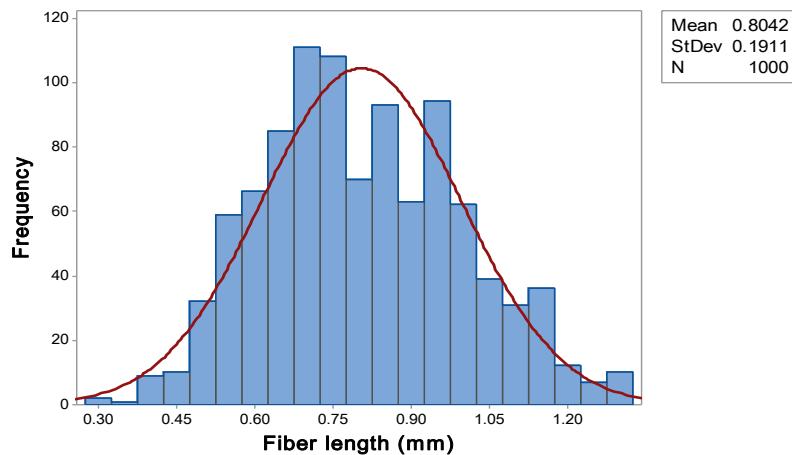
For the heat-treated panels, the analysis of variance showed that all the tested physical properties (WA_{2h}, WA_{24h}, TS_{2h}, and TS_{24h}) were significantly affected by heating temperature, time of heat exposure, and their interaction at 0.05 significance level. The WA_{2h} was found to be 43.56% for the untreated panels while the mean values of the thermal treated panels were 43.54, 43.23, 42.90, 38.10, 36.53, and 34.20% for 180 °C/5min., 180 °C/10 min, 200 °C/5 min, 200 °C/10 min, 220 °C/5 min, and 220 °C/10 min treatments, respectively. The untreated WA_{24h} mean value was 60.28% while the mean values of the thermally treated panels ranged from 54.4% to 60.1%. The lowest value was observed in the 220 °C/10 min treatment. It is worth noting that increasing the heating temperature and exposure time improved dimensional stability. The results showed that the reduction in WA_{2h} ranged from 0.05 to 21.5% for the thermally treated samples compared to the untreated samples, whereas the reduction in the WA_{24h} ranged from 0.3-9.8%.

Fisher's test for WA_{2h} revealed insignificant variations among the untreated panels, 180 °C/5 and 10 min, and 200 °C/5min at 0.05 level of significance. The same results of Fisher's test were also observed in the WA_{24h} except there was a significant difference between the untreated and the 200 °C/5min treatment.

Table 2. Chemical composition of *Ficus retusa* branchwood

Parameter	Chemical composition			
	a- Cellulose	Lignin	Total extractives	Ash
Mean ± standard deviation	47.76 ±1.5	29 ±1	6.76 ±0.91	2.19 ±0.33

Note: Values in percentage.

**Figure 1. Frequency distribution histogram of fiber length of *F. retusa* branchwood.****Table 3. Water absorption (WA) and thickness swelling (TS) of untreated, wax emulsion, and thermally treated particleboards.**

Physical properties	Untreated	Panels with 1% wax emulsion	Temperature					
			180 °C		200 °C		220 °C	
			5 min	10 min	5 min	10 min	5 min	10 min
WA _{2h} (%)	43.56 ^a (0.8)	34.25 ^d (0.1)	43.54 ^a (0.63)	43.23 ^a (0.49)	42.90 ^a (0.44)	38.10 ^b (0.56)	36.53 ^c (0.32)	34.20 ^d (0.4)
Difference	-	-21.4%	-0.05%	-0.8%	-1.5%	-12.5%	-16.1%	-21.5%
WA _{24h} (%)	60.28 ^a (0.5)	53.40 ^f (0.3)	60.1 ^a (0.7)	59.97 ^a (0.31)	59.10 ^b (0.3)	57.17 ^c (0.25)	56.10 ^d (0.2)	54.40 ^e (0.26)
Difference	-	-11.4%	-0.3%	-0.5%	-2%	-5.2%	-6.9%	-9.8%
TS _{2h} (%)	16.44 ^a (0.35)	11.19 ^e (0.20)	16.41 ^a (0.49)	16.21 ^{ab} (0.54)	15.57 ^b (0.47)	14.51 ^c (0.52)	13.07 ^d (0.18)	11.4 ^e (0.15)
Difference	-	-31.9 %	-0.2%	-1.4%	-5.3%	-11.7%	-20.5%	-30.7%
TS _{24h} (%)	29.50 ^a (0.57)	19.16 ^e (0.7)	29.47 ^a (0.7)	29.10 ^a (0.31)	28.21 ^b (0.3)	24.70 ^c (0.25)	21.18 ^d (0.2)	19.93 ^e (0.26)
Difference	-	-35.1%	-0.1%	-1.4%	-4.4%	-16.3%	-28.2%	-32.4%

Means followed by the different letters in the same row for each physical property are statistically different according to Fisher's LSD test at 0.05 % significance level; values in parentheses are standard deviation; the difference (%) for the panels with wax emulsion and the thermal- treated panels is based on the difference from the untreated panels.

Moreover, with the increasing temperature from 200 to 220 °C and heat exposure to 10 min significantly improved both the WA_{2h} and WA_{24h}. H'ng *et al.*, (2011) reported that three heating temperatures (100, 150, and 180 °C) did not affect the water absorption of particleboard. Additionally, the samples treated with wax emulsion (1%) reduced the water absorption for both 2 h and 24 h compared with the untreated samples. For both WA_{2h} and WA_{24h}, the statistical analysis showed that the wax emulsion treated panels varied significantly from the untreated and heat-treated panels while there was no significant difference between the wax emulsion and the heat-treated panels at 220°C/10min for WA_{2h}. The wax emulsion (1%) as an additive reduced significantly the WA_{24h} compared with the 220 °C/10min. treatment.

The thickness swelling of the heat-treated panels ranged from 11.4% to 16.41% and from 19.93% to 29.47% for the 2 h and 24 h of water soaking, respectively. Fisher's LSD test for both TS_{2h} and TS_{24h} revealed no statistical differences among the panels treated at 180 °C for 5 and 10 min. and the untreated panels. The results showed that the reduction in the TS_{2h} ranged from 0.2 to 30.7% for the thermally treated samples compared to the untreated samples, whereas the reduction in the TS_{24h} ranged from 0.1 to 32.4%. The 200 °C/5min., 200 °C/10min., 220 °C/5min., and 220 °C/10min treatments reduced significantly both TS_{2h} and TS_{24h}. In contrast to the findings of the current study, H'ng *et al.*, (2011) reported that three heating temperatures (100, 150, and 180 °C) affected the thickness swelling of particleboard and the greatest effect was observed for the 180 °C treatment. The results showed that the treated samples with wax emulsion (1%) reduced the TS_{2h} and TS_{24h} in comparison with the untreated samples. Fisher's test indicated insignificant differences were found between samples treated with paraffin wax and the 200 °C/10

min treatment; hence, both effects on the TS_{2h} and TS_{24h} are comparable.

Based on the results of this study, the increasing heating temperature and heat exposure time from 200 °C improved significantly all the measured dimensional stability properties. Moreover, the treatment with 1 % wax emulsion had the same effect of 200 °C/10min. treatment on improving the dimensional stability. Carvalho *et al.*, (2015) applied three thermal treatments (200, 230 and 260 °C) on particleboard produced from bagasse and found that water absorption in all the treatments varied significantly from the control. Del Menezzi, *et al.*, 2009 reported that the post-heat treatment using two temperatures (190 and 220 °C) reduced the water absorption and thickness swelling of laboratory-produced oriented strand board. Winandy and Krzysik, (2007) reported that after manufacturing hot pressing improved the dimensional stability of MDF panels. The formaldehyde emission levels from the post-manufacture thermal treated, and wax emulsion *Ficus* particleboards are presented in Table 4.

The formaldehyde emission mean values of the tested panels after five minutes of heat exposure were found to be 2.10, 1.83, and 1.47 mg/L for the 180, 200, and 220 °C, respectively. Whereas, after 10 minutes of heat exposure were 2.0, 1.68, and 1.38 mg/L for the 180, 200, and 220 °C, respectively. The analysis of variance showed that the heating temperature, heat exposure time, and the interaction between them had a significant effect on the formaldehyde emission at 0.05 significance level. Generally, there are classes for formaldehyde emission according to each specified standard. For example, the limits for formaldehyde emission from wood products based on the desiccator method used in this study are classified into three categories: ≤0.3-0.4, ≤0.5-0.7, and ≤1.5-2.1 mg/L, these values are denoted as F****, F***, F**, respectively (JIS A 5905 and 5908, 2003).

Table 4. Formaldehyde emission of untreated, wax emulsion, and thermally treated particleboards manufactured from *Ficus retusa* branchwood.

Untreated	Panels with 1% wax emulsion	Temperature					
		180 °c		200 °c		220°c	
		5 Min.	10 Min.	5 Min.	10 Min.	5 Min.	10 Min.
2.13 ^a (0.04)	2.24 ^a (0.05)	2.10 ^a (0.07)	2.0 ^a (0.13)	1.83 ^a (0.05)	1.68 ^b (0.031)	1.47 ^c (0.03)	1.38 ^c (0.02)
Difference (%)							
5.16%	-1.41	-6.1	-14.1	-21.1	-31	-35.2	

Note: Values in (mg/L); values in parentheses are standard deviation; means with the same letter are not significantly different; the difference (%) for the panels with wax emulsion and the thermal- treated panels is based on the difference from the untreated panels.

These formaldehyde emission classes in the European standards denoted as E₀, E₁, E₂, and E₃. It is worth mentioning that there are conversion factors can be used to compare among the common methods used for formaldehyde emission (Hemmilä *et al.*, 2018). Accordingly, all the determined formaldehyde emission values including the post-thermal treatments and the treatment with wax additive were in the F★★ emission class. It is noteworthy to mention that, in 2010, the World Health Organization (WHO) mentioned a guideline for formaldehyde concentration of 0.08 ppm as a recommended value for short-term exposure (30 min.) in the indoor environment (WHO, 2010). Fisher's test for means comparison showed that there were insignificant variations among the 180 °C/5 and 10min. and 200 °C/5 min. treatments. The data showed that the 200°C /10 min and 220°C/5 and 10 min. treatments were effective in formaldehyde release reduction. Insignificant variation in the formaldehyde emission was found between the untreated and the panels containing 1% wax emulsion at 0.05 significance level. This indicates that the use of wax emulsion (1%) as an additive showed an inability to reduce formaldehyde emission compared to the post-thermal treated panels. Thus, the post-thermal treatment is more effective in reducing the formaldehyde emission than the addition of 1% wax emulsion. Therefore, this study introduces to the particleboard manufacturers a possible way to reduce formaldehyde emissions from their products before introducing them to the market. Moreover, this method may be less expensive than other methods used in reducing formaldehyde emission from wood products. Further studies need to be conducted to examine the total effect of this procedure on the mechanical behavior of these particleboards following the parameters introduced in this study.

CONCLUSIONS

A comparison of the effect of post-manufacture thermal treatment and wax emulsion as an additive on dimensional stability and formaldehyde emission of particleboard produced from *Ficus retusa* branchwood was investigated. Three Temperatures and two-heat exposure time were used for thermal treatment. The statistical analysis revealed that both heating temperature and heat exposure time affected significantly the formaldehyde emission, water absorption, and thickness swelling. The results revealed that the thermally treated particleboard at 200 °C /10 min and 220 °C for 5 and 10 min were the most effective among the other thermal treatments for improving the dimensional stability and reducing

formaldehyde emission. The addition of 1% wax emulsion improved the thickness swelling and water absorption of the panels and was comparable with those thermally treated panels at 220°C/10 min, however, no significant reduction in the formaldehyde emission was observed. On the other hand, the results of the chemical composition and the fiber length of the raw material showed the suitability of *Ficus retusa* branchwood for particleboard manufacturing.

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الملخص العربي

تأثير المعاملات التكنولوجية المختلفة على الخشب الحبيبي المصنع من فروع الفيكس العادي: الخواص الفيزيائية وانبعاث الفورمالدهيد

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٢٣٠ ° م لمنا ٥ دقائق كان له تأثير كبير في تحسين خواص الألواح من حيث الانتفاخ والزبادة في سمك الألواح وكذلك على مستوى انبعاث الفورمالدهيد. بينما وجد ان أضافة مستحلب الشمع بنسبة ١% كإضافة أثناء التصنيع قد حسن من خواص الثبات البعدى للألواح وكان يضافي النتيجة المتحصل عليها من المعالجة الحرارية للألواح على درجة حرارة ٢٠٠ ° م لمنا ١٠ دقائق لم يكن له أي تأثير معنوي على مستوى الانبعاث الفورمالدهيدي. من ناحية آخرى، فإن نتائج التحليل الكيميائى وطول الألياف للمادة الخام المستخدمة في تلك الدراسة أظهرت إمكانية استخدام فروع أشجار الفيكس العادي كمادة خام لصناعة الخشب الحبيبي.

يهدف هذا البحث إلى مقارنة تأثير المعالجة الحرارية وتأثير إضافة مستحلب الشمع على الانتفاخ والزبادة في السمك وكذلك مستوى انبعاث الفورمالدهيد على خواص ألواح الخشب الحبيبي المصنع من فروع أشجار الفيكس العادي الناتجة من عملية تقليم الأشجار. الألواح المصنعة كانت أحادية الطبقية بكثافة نهائية ٦٩٠ كجم/م^٣ واستخدم مستوى غراء يوريا فورمالدهيد ١٢%. تم تطبيق ثلاث معاملات حرارية مختلفة (١٨٠-٢٠٠-٢٢٠ ° م) على فترات زمنية مختلفة (٥، ١٠ دقائق). التحليل الإحصائي للنتائج أوضح ان المعالجة الحرارية على درجة حرارة ٢٠٠ ° م لمنا ١٠ دقائق وكذلك المعالجة الحرارية على