

IMPACT OF PAPER DEINKING SLUDGE AND GLUTARALDEHYDE ON SOME NATURAL FIBER COMPOSITE PROPERTIES

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This study investigated the effects of incorporating paper deinking sludge (PDS) and glutaraldehyde (GA) as an alternative binder in the production of fiberboard. Four types of fiberboards were manufactured: control boards with urea-formaldehyde resin (CUF) and GA resin (CGA), and boards containing 20% (FS20) and 30% (FS30) PDS with GA resin. The mechanical properties (MOR, MOE, IB, and Shore D hardness) of the produced boards were also measured following EN standards. The GA-bonded boards (CGA, FS20, FS30) exhibited lower mechanical performance, but varying flexibility and hardness. The incorporation of PDS improved the mechanical properties compared to the CGA control. The results showed that adding up to 20% PDS content can enhance the mechanical strength of the boards. Additionally, bacterial and fungal growth on the boards was assessed using hygiene test kits. Bacterial and fungal tests revealed that GA resin effectively reduced microorganism viability on the board surfaces compared to the CUF specimen. However, boards with higher PDS content (FS30) showed more microbial growth than those with lower PDS content (FS20). This study demonstrates the potential for utilizing PDS and GA in fiberboard production, offering a sustainable alternative to conventional raw materials and resins.

Keywords: fiberboard, deinking sludge, glutaraldehyde, mechanical

INTRODUCTION

Paper deinking sludge (PDS) is the waste sludge produced during the deinking process, which is one of the pulp cleaning processes involved in the recycling of used paper. About 40–50 kg of dry sludge is produced by the paper industry for each tonne of paper produced, according to Bajpai, ¹Jaria² *et al.*, and Goel and Kalamdhad.³ It is composed of 70% primary sludge and 30% secondary (biological) sludge. For one tonne of newsprint to tissue paper, 170–600 kg of paper deinking sludge (PDS) is produced on a dry mass basis.^{1,4} As a result, solid waste builds up and is disposed of in landfills, raising environmental concerns.^{5,6} In spite of this, PDS has significant potential for use in a variety of industrial and environmental applications due to its composition of cellulose fibers, inorganic fillers, and organic residues.⁷

Research shows that PDS is viable as a feedstock for bioenergy production via anaerobic digestion (biogas) or direct combustion for thermal recovery, providing a sustainable alternative to fossil fuels.^{8,9} In agriculture, processed sludge can

be used as a soil amendment or compost enhancer, improving water retention and nutrient availability while diverting organic waste from landfills.^{10,11} Studies in the construction industry emphasize that PDS can be converted into simple bricks,¹² cement-based composites^{13,14} and acoustic panels¹⁵ due to its pozzolanic properties and low density, enhancing material performance. Additionally, the remaining cellulose fibers from PDS can be recycled into molded pulp, packaging, or inferior paper products.^{16,17} Furthermore, sophisticated processing allows PDS to be incorporated into various composites as reinforcement fillers for the production of plastics, containers, and automotive components.^{18,19} Micronized PDS particles have shown potential as sustainable fillers in asphalt mixtures²⁰ and rubber¹⁸ production, reducing dependency on virgin resources. Research has demonstrated the potential for the enhancement of the utilisation of PDS in wood-based panels. A recent study has indicated that the incorporation of up to 15% paper sludge in the production of particleboard is feasible and does not adversely

affect the quality of the final product, provided that the particle size is meticulously regulated.²¹ The incorporation of 20% PDS content has been proven to enhance the sustainability and circularity of MDF production, thereby motivating the diversion of waste materials from landfills. Furthermore, it has been demonstrated that a 20% PDS significantly reduces raw material costs.²² Moreover, the presence of small sludge particles, measuring less than 1 millimetre, has been identified as a pivotal factor in enhancing mechanical properties, while concurrently reducing water absorption and thickness swelling.²¹

These numerous uses not only promote circular economic principles, but also help to minimize waste, energy consumption, and environmental effects, highlighting the significance of ongoing research into PDS valorisation methods. Recovering PDS in the paper industry is important for sustainable production and waste management. Thus, this study investigated the effects of using PDS as a filler in fiberboard production after drying and grinding it, as well as using glutaraldehyde (GA) instead of traditional urea-formaldehyde (UF) resin. Although formaldehyde-based resins are commonly used in fiberboard manufacture, their carcinogenic and environmental dangers urge the development of more sustainable alternatives.²³

GA is a clear, oily liquid with a pungent odor that mixes readily with benzene, alcohol, and water under standard conditions.²⁴ This chemical has a wide range of uses, such as in water floods, paper mill processing systems, metalworking fluids, water-based conveyor lubricants, air washers, and industrial scrubbers. More specifically, it is applied in oil storage tanks, drilling muds, completion and workover fluids, packer fluids, gas production pipelines, gas storage wells, and related systems.²⁴ In the medical field, glutaraldehyde is commonly used to disinfect non-living surfaces, with concentrations varying based on the formulation.²⁴ Additionally, hospitals utilize antiseptic solutions containing glutaraldehyde (GA) for sterilizing medical equipment due to its ability to inactivate viruses and provide effective sterilization.²⁵⁻²⁷ Moreover, GA significantly improves the bonding performance of protein-based resins by enhancing crosslinking, as seen in wheat protein resins, leading to stronger plywood bonds.²⁸ It also boosts mechanical and thermal properties, as demonstrated in soy protein resins, increasing tensile strength and thermal stability.²⁹

Additionally, GA enhances structural integrity by forming stable protein matrices, ensuring durability under varying conditions.^{30,31} Its compatibility with other crosslinkers, like polyethylenimine, allows for tailored resin formulations,²⁸ while its versatile reactivity enables extensive crosslinking for stable resin production.³² Overall, GA is a preferred choice for industrial resins due to its performance-enhancing properties.

Given its effective crosslinking and disinfectant properties, glutaraldehyde (GA) – a less toxic alternative to conventional binders – was used as a resin in this study, and the mechanical performance of fiberboards containing 20–30% PDS was systematically investigated. The hypothesis is based on the assumption that inserting fine PDS particles into the board composition will improve density and resistance qualities, which is inspired by previous research from existing work on pure fiber-based papers containing fines.³³ The selected PDS concentration range (20-30%) was based on prior research, which demonstrated that this ratio balances waste utilisation with structural integrity, while exceeding 30% sludge has been reported to reduce water resistance and mechanical strength significantly.³⁴ Geng *et al.*³⁴ demonstrated that boards containing 30% PDS met the standards; however, adding more than 30% PDS reduced the mechanical strength to a critical level. This is because the high ash content and short fiber structure of PDS weaken the bonding between the fibers. Specifically, parameters such as internal bond (IB) and bending strength (MOR) decreased linearly as the PDS percentage increased. Using 20% PDS was found to be an optimal threshold that balances waste content with mechanical integrity. Regarding water resistance, the inorganic structure of PDS improves thickness swelling (TS) to some extent; however, the degradation of the fiber matrix at high rates eliminates this advantage. Similarly, the other studies on the use of PDS in cement mortar products demonstrated that it could absorb significant amounts of water in the product mix.^{14,35} Therefore, additional solutions, such as adding wax or alternative binders (*e.g.*, glutaraldehyde), are required for using PDS in board production.

The goal of this study has been to test the binding efficiency of PDS-containing natural fiber-based composites, aligning with fibers, while taking advantage of GA's high reactivity and crosslinking capacity. Mechanical tests and hygienic analyses were used to examine the effects

of PDS content and resin type, which shed light on the viability of producing fiberboard.

EXPERIMENTAL

Materials

The fiber furnishes consisted of 80% beech and 20% larch wood fiber mixtures, and the wood fibers used in the production of the laboratory-made board were supplied by Kastamonu Entegre MDF Gebze Factory (Kocaeli, Türkiye) in their natural state, without additives. Ammonium sulfate ((NH₄)₂SO₄) hardener and urea-formaldehyde (UF) resin were supplied by Yıldız Entegre Akhisar Facilities (Manisa, Türkiye). The F/U mole ratio of 1.15, density of 1.28 g/cm³, pH

of 7.91, and viscosity of 84 cps were all recorded for the UF resin with 64.93% solid content at 23.4 °C. Alfasol glutaraldehyde chemical (GA 50%) was acquired commercially. Paper deinking sludge (PDS) was supplied as a waste from the Europap Tezol Izmir Paper Mill deinking line. The paper deinking sludge had an average density of 0.95 g/cm³ and a moisture content of 3.40% at room temperature.

Figure 1 presents the provided paper deinking sludge (PDS), ground PDS and wood fibers utilized in the production of boards. The moisture content of PDS and wood fibers was reduced to 3–5% before board was produced. To prevent the moisture from interacting with the surroundings, the fibers and PDS were sealed in plastic bags.

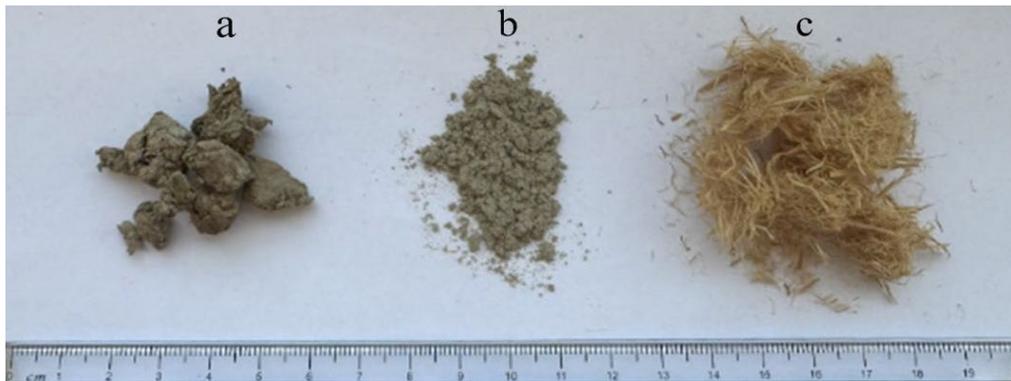


Figure 1: a) PDS as received, b) ground PDS and c) wood fibers used in board production

Grinding of PDS

A laboratory-type 40 mesh sieve grinder was used to grind the paper deinking sludge to about 400 µm in size, so that it could be used in board production as a raw component. It was hypothesised that reducing size and sieved PDS particles to a fine 40-mesh size and incorporating them at 20–30% of the board composition would enhance the board's density and resistance properties. This was based on the theory that using 10–20% fines in paper material increases the density and mechanical resistance values.³³

Preparation of glutaraldehyde (GA) binders

A 50% concentration of GA was combined with room temperature pure water to achieve a 25% concentration. The binder solution for the boards was then prepared by adding 1% of its weight of ammonium sulfate hardener, and all components were combined using a SpeedMixer set to 500 rpm for five minutes. A Brookfield type viscometer, with a three-levelled mixer and a speed range of 50 and 100 rpm, was used to measure the viscosity values of the UF resin and GA binders at a temperature of 22±1 °C.

Board manufacturing

The fiberboards were produced using varying wood fiber-to-PDS ratios (100/0, 70/30, 80/20 wt/wt). Figure 2 illustrates the manufacturing process for single-

layered fiberboards composed primarily of wood fibers with partial PDS incorporation. The target board specifications were 9 mm thickness and 750 kg/m³ density, achieved using a laboratory-scale hydraulic hot press (Cemilusta SSP-180 T Model, Istanbul, Türkiye). The board production process flow chart is given in Figure 2. Two binder types were employed: (1) glutaraldehyde (GA) binder and (2) urea-formaldehyde (UF) resin – the latter being standard in industrial production. The UF resin was used exclusively for one control board (CUF), while GA was applied to the remaining control board (CGA) and PDS-containing boards (FS20 and FS30). The manufactured board contents and their sample codes are given in Table 1. Both binder types were homogenized using a mechanical mixer (30 rpm) and applied at a fixed binder content (15 wt%). Board mats were manually formed in a 320 × 360 mm mold and subsequently hot-pressed at 170 °C for 5 min under 3 MPa pressure. Representative samples of the produced boards are shown in Figure 3. Prior to testing, all boards were conditioned for two weeks at 20 °C and 65% relative humidity.

Physical and mechanical testing

The boards' mechanical characteristics were assessed following their cutting to the required test size in accordance with European Norm (EN) guidelines. The EN 317³⁶ standard was followed in determining the

board samples' water absorption capacity. The EN 310 standard³⁷ was used to determine the mechanical properties' bending strength (MOR) and modulus of elasticity (MOE), while the EN 319 standard³⁸ was used to determine the internal bond (IB) strength. The tests were performed using an IMAL IB600 Universal Testing Machine.

The Shore D hardness tests were carried out according to ASTM D2240³⁹ standard. For the mechanical tests, ten test specimens of each kind of board were utilized.

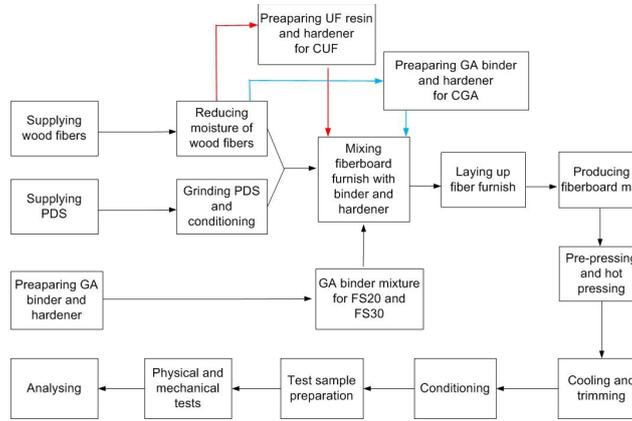


Figure 2: Board manufacturing process

Table 1
Codes of board grades and their composition

Board code	Composition of the boards (%)			
	Wood fiber	PDS	Binder*	Hardener*
CUF	100	-	UF resin	(NH ₄) ₂ SO ₄
CGA	100	-	Glutaraldehyde	(NH ₄) ₂ SO ₄
FS20	80	20	Glutaraldehyde	(NH ₄) ₂ SO ₄
FS30	70	30	Glutaraldehyde	(NH ₄) ₂ SO ₄

* The binder accounted for 15% of the board weight, while the hardener, ammonium sulphate, made up 1% of the binder weight

Bacterial and fungal tests

All types of produced boards were examined for bacterial and fungal activity using microbial samples found on the surface of unwashed lab workbenches and human hands. The Hytech Slide® hygiene test kit was used to track the growth of bacteria and fungi on the board surfaces. Each Hytech Slide® has two medium sized surfaces. Each surface is divided into ten 1 cm² areas. Following five to six seconds of contact with the growth medium in the test kits, all coded samples were stored in an oven at 35 °C for 48 hours for bacteria and in a conditioned test room at 25 °C for 120 hours for molds and fungi. The results were expressed as the number of colonies and microbial density in a surface area of 10 cm².

RESULTS AND DISCUSSION

Characteristics of boards and binders

Representative images of the four types of laboratory-made fiberboards are shown in Figure 3. In order to comprehend the PDS effect, control boards made with UF resin (CUF) and GA binder

(CGA) were compared to boards that had 20–30% PDS additive (FS20 and FS30). The measured viscosity values of the prepared glutaraldehyde (GA) binder and urea formaldehyde (UF) resin are listed in Table 2 as a function of temperature and two-stage rotor speed.

Mechanical test results

Table 3 shows the mean values of thickness swelling (TS) and water absorption (WA) measurements of board specimens taken after 2 and 24 hours of submersion in water.

Based on the EN 317 (1999) standard, optimal dimensional stability was demonstrated by the CUF board specimens. According to the data in Table 3, the board is more or less stable and most swelling occurs in the first two hours of water contact, with small further swelling up to 24 hours (32.65% to 37.10%). This suggests that urea-formaldehyde (UF), a water-resistant resin, is very effective in ensuring board stability. Meanwhile,

the specimens of CGA boards show extremely low long-term stability. They swell significantly in 24 hours (66.70%), even though their 2-hour TS is the lowest (22.20%). This denotes a structure or binder that initially resists water, but eventually fails dramatically, most likely because of severe capillary action or binder deterioration. Specimens FS20 and FS30, formulated with GA and PDS, exhibited exceptionally high 24-hour

thickness swelling (TS) values of 88.90% and 77.82%, respectively.

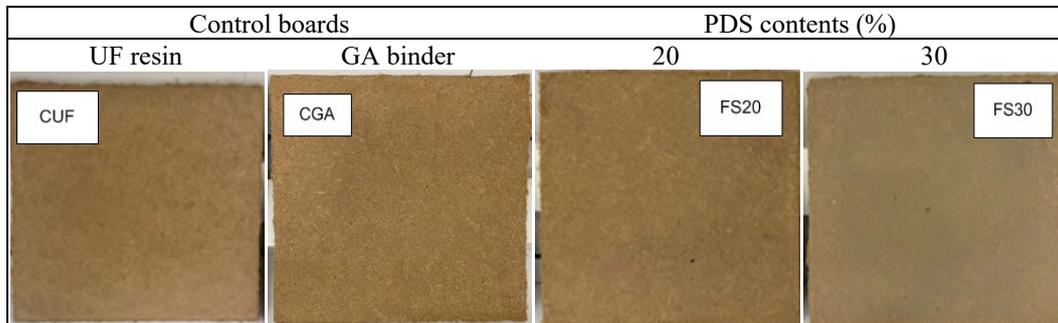


Figure 3: Four types of representative fiberboards produced in the laboratory

Table 2
Measured viscosity values of UF resin and GA binders

Binder type	Temperature (°C)	Rotor speed	
		50 rpm (cP)	100 rpm (cP)
UF	22±1	27	18
GA	22±1	35	24

* Binder with hardener, ammonium sulphate (1% of binder weight)

These values substantially exceed the acceptable threshold for standard interior-grade boards, indicating poor moisture resistance. This is likely attributable to the hygroscopic nature of the raw material and the GA-based binder. Although the higher PDS content in FS30 (30% vs. 20%) appears to confer slightly improved stability, the effect is insufficient to meet required standards.

The water absorption (WA) values of the boards (in Table 3) are indicative of board porosity and hydrophilicity. The CUF board shows rapid initial

absorption (73.92% after two hours), followed by a minimal increase, which suggests quick saturation and consistent dimensional stability. In contrast, the CGA board exhibits moderate initial water absorption (WA) followed by a significant increase after 24 hours. The FS20 and FS30 boards demonstrate extremely high WA at both intervals, especially the mean values of FS20 specimens exceeding 116%. These data confirm the high porosity and inherent hygroscopicity characteristics of these boards, which directly correlate with their poor moisture resistance.

Table 3
Mean values of TS (2 to 24 h), and WA (2 to 24 h) of the boards

Board code	2 h TS (%)	24 h TS (%)	2 h WA (%)	24 h WA (%)
CUF	32.65	37.10	73.92	83.71
CGA	22.20	66.70	52.26	65.36
FS20	44.35	88.90	102.72	116.32
FS30	34.39	77.82	77.18	96.57

Table 4
Mean values of mechanical properties of the boards

Board code	MOR (MPa)	MOE (MPa)	IB (MPa)	Shore D Hardness
CUF	31.32 (2.70)	2837.64 (207.80)	0.23 (0.03)	70.00 (5.83)
CGA	2.97 (0.31)	165.46 (47.75)	Not applicable	57.60 (6.86)
FS20	3.48 (0.27)	712.05 (119.53)	Not applicable	47.95 (6.02)
FS30	3.57 (0.43)	556.97 (175.91)	Not applicable	65.90 (5.21)

Values in parentheses are standard deviations

Table 4 presents material performance and potential applications by comparing the mechanical properties (MOR, MOE, IB, Shore D hardness) of four distinct boards. Standard deviations show the consistency of the measurements.

The CUF board demonstrates greater mechanical performance compared to the other boards containing GA and PDS, with an MOR of 31.32 MPa and an MOE of 2837.64 MPa. These mechanical characteristics enable the board to meet the minimum requirements specified in the European Standard EN 622-5 (2009)⁴⁰ for non-load-bearing boards for use in both dry and humid conditions, as well as for load-bearing boards used in dry conditions. Therefore, the CUF board is well-suited for structural applications. On the other hand, because of the use of GA binder, the CGA control board shows the lowest mechanical performance with an MOR of 2.97 MPa and an MOE of 165.46 MPa. The MOR and MOE values of the boards increased by 17.17% and 330.35%, respectively, with the addition of 20% PDS (FS20). However, when 30% PDS (FS30) was added to the board content, the MOE value decreased by 21.78%, although the MOR value increased by 2.59% compared to the FS20 board type. It can be explained by the fact that PDS often contains shorter, defective and damaged cellulose fibers, which reduce the mechanical strength of the boards.⁴¹ Migneault *et al.*⁴² reported that incorporating up to 25% PDS content can enhance the mechanical strength of the boards, making the resulting fiberboards suitable for interior applications.

Shore D hardness values for each board type were established. This test evaluates the surface hardness of a material by determining its resistance to indentation (ASTM D2240).³⁹ Shore-D hardness varies between 47.95 and 70.00. It is clear that the surface hardness decreased with the addition of GA and PDS in the fiberboard. The results obtained show that the PDS additive affects the hardness in

a non-linear manner depending on the content ratio. The IB value for the boards containing GA and PDS was not applicable. Kim *et al.*⁴³ explained that the presence of inorganic substances like kaolin clay and calcium carbonate in PDS adversely affects the interfacial bonding with wood fibers.

Bacterial and fungal test results

The CUF, CGA, FS20, and FS30 board specimens were subjected to bacterial and fungal test using HyTech Slide® hygiene test kits. The microorganisms that developed on the boards were transferred to the agar in the hygiene kits. In accordance with the test kits' user manual, the test kits incubated for five days and inspected to track bacterial and fungal activity development. The yellow-colored medium surfaces were used for bacterial detection, and the pink-colored medium surfaces were used for mold and fungal detection. The bacteria were incubated at 35 °C, and the changes were evaluated at the end of the first, second, and fifth days. Fungi yeasts were incubated at 25 °C, and changes were tracked at the end of the first, second, and fifth days. Figures 4 and 5 show images of the kits displaying changes in bacterial and fungal activities over time.

Antibacterial testing was conducted using HyTech Slide® kits for all four types of boards. The surface of the test kits was subdivided into 10 areas, each measuring 1 cm², and the number of bacteria present on these areas was counted at the end of first, second, and fifth days, respectively. At the end of the fifth day, the count revealed that the number of live bacteria was active in 9 units of the 10-unit area divided for the CUF specimen. On the other hand, during the given time period, the number of live bacteria was active in two units of the 10-unit area divided for the CGA specimen (Fig. 5). The results from CUF and CGA boards showed that the antibacterial properties of GA binder were effective in reducing bacterial viability on the surface of the control boards. The same

effects were observed in the FS20 and FS30 specimens made with GA binder; fewer live

bacteria were active in 2 or 3 units of the 10-unit area divided for these kits as well.

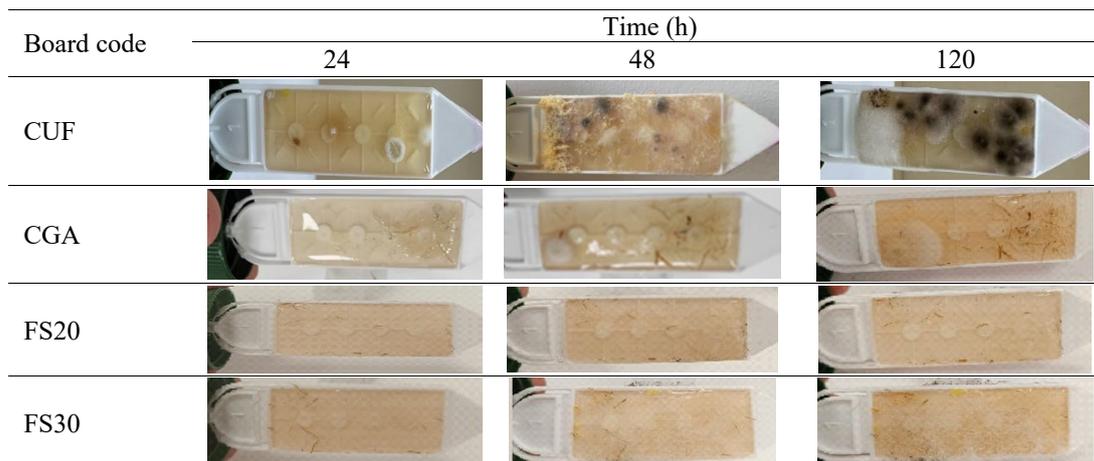


Figure 4: Bacterial growth dynamics on HyTech Slide® kit media over 1, 2, and 5 days of incubation at 35 °C

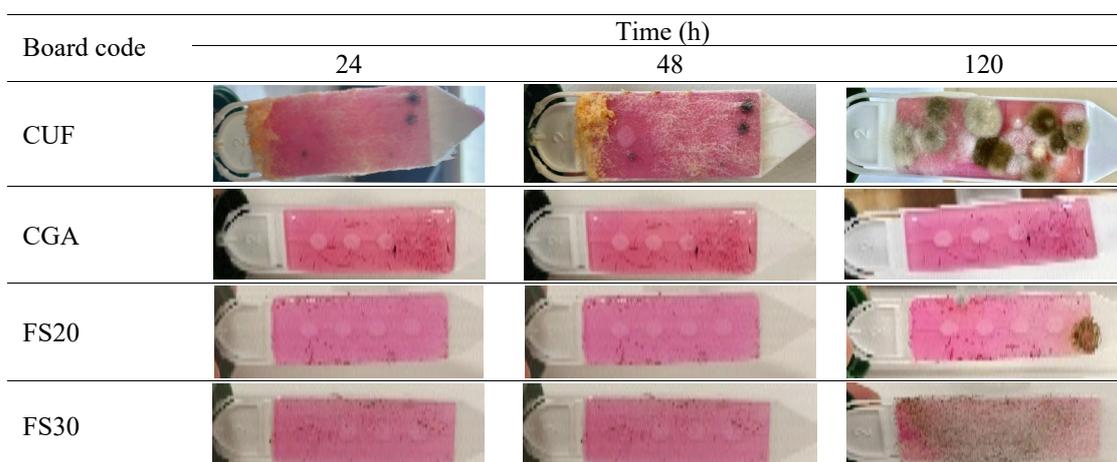


Figure 5: Fungal growth dynamics on HyTech Slide® kit media over 1, 2 and 5 days of incubation at 25 °C

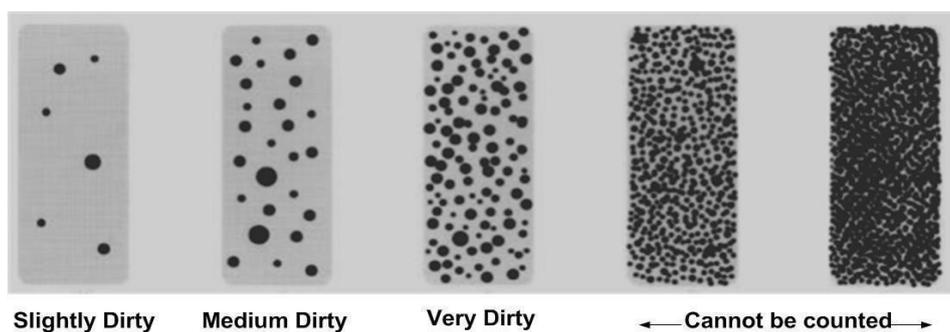


Figure 6: Reference image that assesses the density of microorganisms in a given area

Figure 6 shows the visual grading as described in the Hytech Slide® test kit user manual. According to this grading system, the results were not only ascertained by counting, but also assessed in terms of the microorganism density at given medium areas of 10 cm².

After the incubation periods, microorganism activity with an uncountable density was observed

on all test kit surfaces that belonged to the CUF sample. Samples taken from other boards (CGA, FS20 and FS30) surfaces did not show such high levels of microorganism activity as the ones belonging to CUF. This implies that when GA is utilized as a board binder, it has an antiseptic effect on board surfaces. It was found that samples taken from the surfaces of boards containing 30% PDS

(FS30) consistently contained more microorganisms than those containing 20% PDS (FS20) when the boards containing two different amounts of PDS were compared. This can be explained by the fact that despite the use of dried, ground PDS and of the high temperature applied during board pressing, the PDS may still contain live microorganisms.

CONCLUSION

Bacterial and fungal tests were conducted on CUF, CGA, FS20, and FS30 board specimens using HyTech Slide® hygiene test kits. The tests revealed that the antibacterial properties of GA binder effectively reduced bacterial viability on the surface of the control boards (CGA) compared to the CUF specimen. Similar effects were observed in FS20 and FS30 specimens made with GA binder. Microorganism activity was uncountable on CUF sample test kits, while other samples (CGA, FS20, and FS30) showed lower levels. Boards containing 30% PDS (FS30) consistently contained more microorganisms than those with 20% PDS (FS20), possibly due to live microorganisms still present in the PDS despite the high temperature applied during board pressing.

According to the EN 317 standard, CUF boards, thanks to their UF resin content, offered more reliable water resistance and optimum dimensional stability compared to other boards. On the other hand, FS20 and FS30 boards with GA binder had very poor moisture resistance and high water absorption due to their intrinsic hygroscopicity and porosity, while the CGA board showed significantly high swelling at the end of the testing process. The performance of the boards is directly related to the hygroscopic nature of their materials and the effectiveness of their binder types.

Based on the mechanical properties, it was observed that the use of GA as a binder in the board seriously reduces the mechanical properties of the board. However, an increase in the mechanical properties of the board was observed with the addition of PDS in the board; especially, the MOE value increased by 330% compared to the control board. This can be explained by the fact that GA binder can serve as an effective crosslinking agent for wood fibers and PDS, as it does with proteins and polysaccharides, enhancing material properties and mechanical strength through covalent bonding. Collectively, these findings suggest that GA could be a viable option for modifying PDS to produce boards with higher MOE values. However, attention should be given to optimizing

concentrations and conditions to mitigate side effects, such as a decrease in IB values.

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