Application of Pyrolysis Acid from Date Palm Waste as Wood Preservative

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Matched sets of clear specimens of poplar and pine wood were treated with 3, 5, and 8% (v/v) aqueous solutions of slow pyrolysis acid (PA) derived from date palm waste, to evaluate the effectiveness of PA in controlling mold growth on species of wood. Chemical compounds available in PA were identified by gas chromatography-mass spectrometry analysis. Treated and control specimens were contaminated with *Aspergillus* sp., then incubated in accordance to AWPA E24 (2017). Percentages of occupied surface by mold on each of control and treated specimen with respect to its total surface were evaluated by Image-J software. Comparisons of mean values of these determined ratios and physical examinations showed that, depending on species, the 5 and 8% water solutions of PA prohibited *Aspergillus* growth on tested woods. Experiments were also conducted on a matched set of small clear specimens of beech wood, impregnated by 3, 5, and 8% water solutions (v/v) of PA, using full-cell process and contaminated with decay fungus (*Trametes versicolor*), then incubated. Specifications of the EN 113 (1996) standard were followed. Weight loss was applied for comparing control and treated specimens. Average values of weight losses indicated that the 5% solution was effective, but 8% solution presented a better result.

Keywords: Palm groove; Waste; Pyrolysis acid; Organic preserver

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INTRODUCTION

With 250,000 ha of date palm grooves, Iran is one of the major date producers in the world. High quality date is harvested from approximately 40% of these groves. About 15 dried rachis are trimmed or pruned from each palm tree every year. The statistical figure of the weight of trimmed rachis (fronds) is around 2,200,000 tons (Ahmadi *et al.* 2015). Enormous waste is produced during this time, and the weights of slashed tree trunk or stem scattered through groves must be added. Thus, these two items will result in a large amount of lignocellulosic wastes.

Thus far these wastes are not properly utilized, while they could be used for producing organic pesticide and biochar through slow pyrolysis. Date farmers disperse them and burn them in the groves, but they are not evenly distributed, which results in piles of ash on the soil.

Date palm (*Phoenix dactylifera*) residues are fibrous materials. In recent years, the properties and elemental composition of these wastes were studied on a laboratory scale (*i.e.*, Tables 3, 4, and 13 in Jonoobi *et al.* 2019). These studies were made with the purpose of replacing wood in its fiber-based composites, such as various panels, structural composite lumber, or paper products, because their fibrous nature and fabricated experimental specimens have shown promise (Jonoobi *et al.* 2019). It must be noted that workability of date palm residues is not like other wood species, including the shearing parallel-to-grain and cleavage, which are comparatively high in these wastes, particularly in rachis. Conventional adhesive (urea formaldehyde) that is used for producing wood-based panels does not cure on these wastes' bark-containing particles as well as it does on wood particles, because their barks are siliceous. Thus, the material ought to be debarked or defibrated, which is an energy-consuming process.

An alternative solution would be replacing the adhesive with an expensive type. The fronds (rachis) are tough substances when compared with woods. They are considered to be high tool wearing materials. In view of replacing wood with these wastes in wood-based productions, field-scale studies on alloy of crushing blades need to be conducted. Additionally, comprehensive industrial studies are required related to the manufacture of fiber-based products out of these wastes with common adhesive, in terms of exploring environmental pollution and mechanism of the impact of such products on human's living environment. Performances of these types of composites are not well known yet, and bringing formaldehyde through inclusion of an adhesive to the living environment of humans is not recommended. Many industrial installations with the purposes of profitability and job openings have been established, but later on they were recognized as causes of environmental problems and global climate change.

An important point related to producing fiber-based products out of date palm wastes is the continuous removal of these residuals from date palm groves. This action in the long run will degrade groves' soils with respect to their fertility and nutrient balance. Such results in other cultivation areas about crops residuals have been substantiated (Prasad and Power 1991).

Fortunately, during the last two decades the concept of conversion of crop biomass through thermochemical process (pyrolysis) into a substance that could be returned to soils under cultivations, has been developed. This form of biomass application has been extended to date palm wastes as well. Many useful research studies on topics such as: properties of biochar (Ronsse *et al.* 2013; Gai *et al.* 2014; Jouiad *et al.* 2015; Bensidhom *et al.* 2018), cases of its use as green sorbents of organic and inorganic pollutants (Usman *et al.* 2015, 2016; Al-Wabel *et al.* 2019), improving soil fertility, upgrading and remediating degraded soils (Beesley *et al.* 2011; Hussain *et al.* 2014; Al-Wabel *et al.* 2019), climate change mitigation (Woolf *et al.* 2010; Skjemstad *et al*. 2002), *etc.*, have been published. In all of these studies the functions of biochar were evaluated positively with no doubt. Conducting pyrolysis process is not harmful for the environment and would not cause any net greenhouse gas emissions. Pyrolignous gases in pyrolyzing biomass are condensable by a proper designed system (Hussain *et al.* 2014).

The other pyrolytic product is liquid that results from condensing volatiles that emit from pyrolyzing reactor. This liquid in recent studies is called pyrolysis acid (PA) (Hussain 2015), while it contains several organic applicable chemicals. Thus, to identify these compounds, a deliberate characterization of this PA is needed.

This study was conducted to characterize PA from date palm waste as a wood preservative. The main objectives in this investigation were to evaluate the preserving potential of PA on two species of wood (poplar and pine) against mold growth and beech wood against rotting fungus.

EXPERIMENTAL

Materials

In this research, date palm waste samples were collected from palm grooves in Lamerd (southern territory of Fars province of Iran). Test materials were air-dried to 10% average moisture content, then chipped into particles that measured 10 to 15 mm in length and up to 5 mm in thickness. Then, the prepared waste particles were pyrolysed in a laboratory-scale pilot electrical furnace that was designed by others with a temperature level up to 350 °C (Fig. 1). The furnace was equipped with programmable control panel for time-based temperature. Temperature increment was set as 5 °C·min^{-1}. The weight of test load for pyrolysis ranged from 500 to 1000 g. Total residential time was 5 h. Emitted volatiles from furnace were transferred through steel pipe to a condensing system, which comprised small diameter sloped spiral stainless steel and being bedded in mixture of ice cubes and water at temperature around 0 °C. Condensed PA was collected in a transparent bottle with liquid and gas tight lid. At the end of predetermined residential time (experimentally determined by stopping oil droplet falling into bottle), power was switched off and as a precautionary measure the apparatus was allowed overnight to cool off. Then, the furnace was unloaded, and weights of biochar and PA were determined.

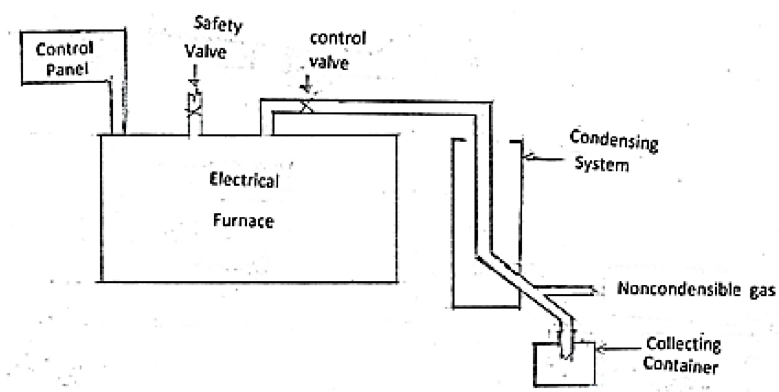

Fig. 1. Schematic diagram of pyrolysis apparatus

Methods

PA analysis

The liquid-liquid extraction was carried out by adding ethyl acetate (Merck, Darmstadt, Germany). Next, 1 mL of the organic fraction was transferred to a gas chromatography vial and was analyzed by gas chromatography-mass spectrometry (GC/MS) using an Agilent 7890A gas chromatograph (Agilent Technologies, Santa Clara, CA, USA) coupled to an Agilent 5975C VL MSD mass spectrometer. The GC separation was performed on a Rtx 5 MS capillary column with a length of 30 m, internal diameter of 0.25 μm, and a film thickness of 0.25 m.

As mentioned earlier in the objectives of this study, it was proposed to investigate the ability of PA in preventing mold growth on solid wood items and also controlling a damaging fungus in a susceptible wood species.

Characterization of PA as wood preservative

Two species of wood, namely poplar (*P. deltoides*) and pine (*P. eldarica*) were treated with the PA derived from pyrolysis date palm waste and contaminated by mold

(*Aspergillus niger*), after being incubated for a two-week period the evaluation of mold's growth was determined by application of the software image-J (National Institutes of Health, Bethesda, MD, USA).

For testing the effectiveness of PA in protecting wood against damaging fungus, beech wood (*Fagus orientalis*) was nominated, because wood of this species is mostly damaged by *Trametes versicolor*.

Mold growth test

Four sets of matched specimens were prepared from each species. Dimensions of each specimen were 4 × 1 × 2 cm^3 (length × thickness × width). Specimens had 10% moisture content. One set of specimens of each species was considered as control agent, the second set of specimens was impregnated by 3% (by weight) water solution of PA, the third set by 5%, and the last set by 8% solution through a full-cell process. Through testing, it was observed that PA was completely water miscible.

All four sets of specimens of individual species were contaminated by *Aspergillus* (mold) and incubated under standard conditions for two weeks (AWPA E 24 2017). After the mentioned incubation period, the percentage of surface in individual specimens occupied by the mold with respect to its total surface was evaluated using image-J software.

Control of decay fungus

Following the same procedure as in the mold test, four sets of specimens from beech wood (*F. orientalis*) were prepared for testing. Every set of specimens consisted of 6 specimens, each with dimensions of 50 × 2.5 × 1.5 mm^3 (longitudinal × radial × tangential) and 10% moisture content. The first set was not treated as control, but the second, third, and fourth sets were impregnated with 3, 5, and 8% (by weight) water solution of bio-oil, respectively, by applying conventional full-cell process. The initial vacuum for 10 min (-0.7 bar) was applied to remove air from wood cavities. Then, the impregnation solution was introduced to the laboratory-scale vessel. Thereafter, the pressure was increased up to 5 bar and held for 2 h. Finally, 5 min vacuum was applied at the end of the process. The control set was soaked in water to saturate. Grown pieces of fungus (*Trametes versicolor*) were placed on the specimens, and then they were incubated for 16 weeks under desired condition of temperature and humidity for fungus growth, carried out in accordance with EN 113 (1996) standard. All specimens were weighed at the end of conditioning period. The weight losses of specimens with respect to their weight after impregnation were calculated.

RESULTS AND DISCUSSION

Results of this process revealed major changes in compositions, as shown in Table 1. Table 1 shows that PA contained six predominant compounds, namely acetone, acetic acid, furfural, phenol, and 1,2-benzenedicarboxylic acid, 3-nitro. Among these compounds, acetic acid has a higher percentage of availability in GC-MS analysis. These six compounds were bioactive in controlling growth of mold and decay of fungus on tested wood species. The pyrolysis derived PA out of oil palm stem functions as an antifungal and antitermitic agent at same time (Oramahi *et al.* 2018).

Table 1. Identified Compositions in PA

No.	Retention Time (min)	Compound Name	Area (%)	Mol. Weight (g/mol)
1	1.601	Acetone	2.03	58.042
2	1.71	Methyl acetate	15.55	74.037
3	2.428	Acetic acid	52.30	60.021
4	5.781	Furfural	17.58	96.021
5	8.994	Phenol	5.89	94.042
6	26.451	1,2-Benzenedicarboxylic acid, 3-nitro-	6.65	211.012

Average retentions of PA in poplar specimens treated by 3, 5, and 8% were calculated as 0.286, 0.336, and 0.965 g, respectively. In pine specimens, in the same order the PA retentions were determined as 0.123, 0.296, and 0.402 g.

Table 2. Data of Mold Growth Experimentation on Poplar and Pine Wood Specimens Treated with PA with Tentative Statistical Measure

Source	Sample Set	No.	Initial Weight (g)	Weight After Treat-ment (g)	Retention (% of Initial Weight)	% Weight of Oil in Specimen		% of Occupied Surfaces to Total Specimen Surface	
						Individual Speci.	Ave.	Individual Speci.	Avg.
Popu-lus	Control	1	10.80					25	23.66
		2	9.62					20	
		3	11.06					26	
	Treated with 3% solution	1	10.23	16.07	57.08	0.175	0.286	14	12.66
		2	10.55	18.85	78.67	0.248		11	
		3	10.39	24.91	139.74	0.435		13	
	Treated with 5% solution	1	10.95	19.29	76.16	0.417	0.336	6	4.66
		2	9.61	15.65	62.85	0.302		3	
		3	9.59	15.23	58.81	0.282		5	
	Treated with 8% solution	1	9.98	23.23	134.62	1.07	0.965	< 5	4.33
		2	9.71	24.56	152.93	1.19		3	
		3	10.44	18.47	76.91	0.642		5	
Pine	Control	1	5.20					18	23.66
		2	5.18					27	
		3	5.39					26	
	Treated with 3% solution	1	5.23	8.80	68.26	0.107	0.123	15	12.33
		2	5.35	8.62	61.12	0.098		8	
		3	4.68	10.16	117.09	0.164		14	
	Treated with 5% solution	1	5.13	10.75	109.55	0.281	0.463	2	3.66
		2	5.22	11.26	115.70	0.302		3	
		3	5.16	11.29	118.79	0.806		6	
	Treated with 8% solution	1	5.16	8.93	73.06	0.801	0.569	1	2.33
		2	4.75	10.34	117.68	0.447		0.0	
		3	4.72	10.46	121.61	0.459		6	

Results of this investigation on mold's growth prevention by applying pyrolysis date palm waste derived PA are presented in Table 2 and Figs. 2 and 3.

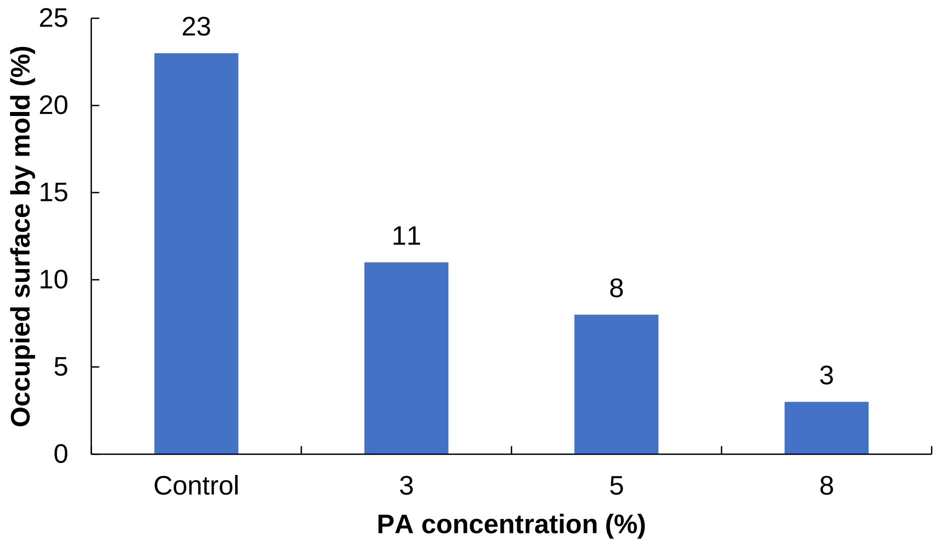

Fig. 2. Occupied surface of specimens by mold (%) on poplar treated and untreated wood

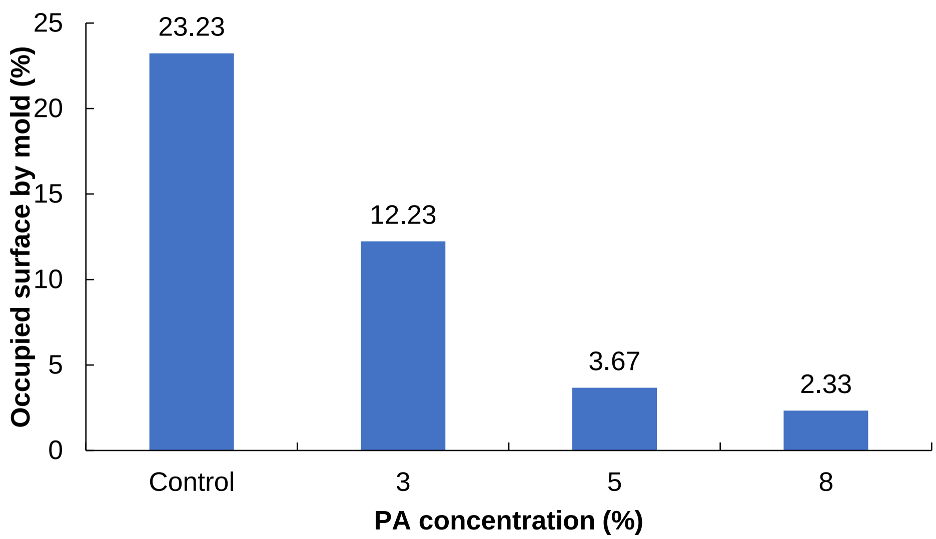

Fig. 3. Occupied surface of specimens by mold (%) on pine treated and untreated wood

Freshly sawn lumber contains saps, which are nutrients for molds and fungi. Molds are not able to damage structure of wood, but dark pigment in their spores will discolor wood (blue, dark, or green) (Ebrahimi 2011; Reinprecht 2016). Discolorations reduces economical values of lumber in their marketplace. The economic importance of preventing stain in wood is so important that saw mills and kiln dryer owners in North America have referred to the Forest Products Laboratory (USDA) for developing a technique using any chemicals that can inhibit mold growth on green lumber. Initial trials revealed that reducing moisture content of lumber to below 20% is a useful action (Ebrahimi 2011). Stacking green lumber after being sawn helps but may not provide a sufficient drying rate, particularly in humid regions where relative humidity mitigates drying rate and mold agents will find the chance to initiate their growth and expand their settlement on lumber. Accelerating air circulation through stacked lumber works to some extent in lowering moisture content below susceptible level for molds. Even applying these types of prohibitive care may not serve the purposes in some places where high relative humidity is present all year around.

Continuation of investigating efforts has found that sodium pantaclorophenate and ethyl phenate mercury can prevent mold growth quite effectively (Ebrahimi 2011). These chemicals were in use for several years until it was recognized that both of them are harmful for workers who work in mills and also these compounds contaminate run-off sources of water. Therefore, the Environmental Protection Agency (EPA) has prohibited uses of these chemicals in 1970 (Ebrahimi 2011). Since then, interested researchers have focused on the usage of organic compounds to meet these objectives (Qu *et al*. 2019).

In this study, the effectiveness of pyrolysis date palm waste derived PA for prevention of mold (*Aspergillus*) growth on poplar and pine wood specimens was evaluated by observing the percentage of the ratio of occupied surface by mold to total surface of specimen as comparative measure. Evaluation of this criterion was made using image-J software. Table 2 contains the details of collected experimental data. The last two columns of this table show values of mentioned criteria over individual control and treated set of specimens. Mean value for control set of poplar specimens was 23.66 and for those of treated ones (3, 5, and 8%) the values were 12.66, 4.66, and 4.33, respectively. In the same order for pine specimens these were 23.66, 12.33, 3.66, and 2.33. Figures 2 and 3 present these values graphically. The physical status of experimental specimens is shown in Fig. 4. It was evident that mold was not able to exhibit its varying intensity of discoloration on specimens of poplar and pine treated with 5 and 8% of solutions (the last two columns of specimens in Fig. 4). The 3% solution on pine wood was more effective than on poplar.

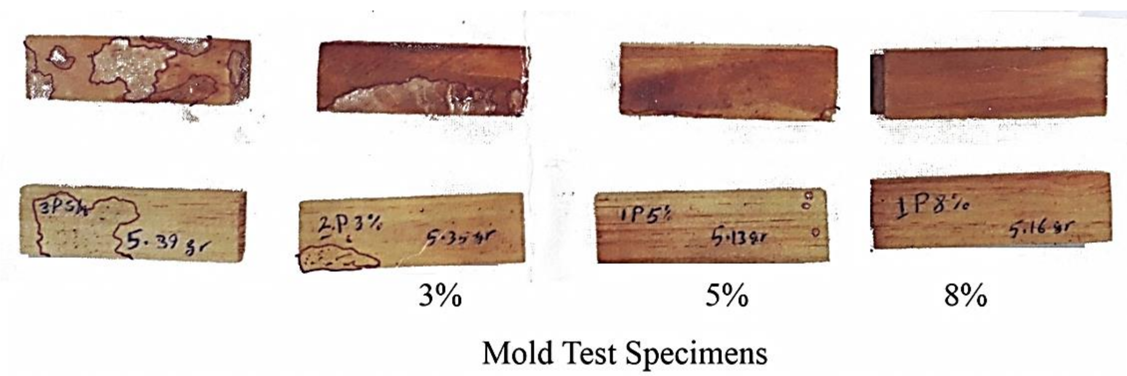

Fig. 4. Physical status of mold contaminated samples after incubation: above row is pine sample, and below one is poplar.

Weight losses of specimens after the decay resistance test are presented in Table 3 and Fig. 5.

The effectiveness of pyrolysis-derived bio-oil in preserving beech wood against fungus (*T. versicolor*) has been characterized through weight reduction between control set and impregnated set of specimens. After treating with solutions that contained 3, 5, and 8% of bio-oil (by weight), the weight reductions of specimens were recorded, and the weight after treatment was considered for comparison measures. The obtained data are listed in Table 3. The last two columns show the percent of weight reduction of individual specimens in each set and their corresponding mean values.

Table 3. Data of Experiment of Decay Fungus on Beech Specimens Treated with PA

Specimen Set	Specimen No.	Initial Weight (g)	Weight After Treatment (g)	Retention, % of Initial Weight		Net Weight of Oil in Samples (g)		% of Weight Loss Based on Weight After Treatment	
				Indiv.	Avg.	Ind. Speci	Avg.	Indiv.	Avg.
Control	1	1.88						12	7.90
	2	1.96						7.20	
	3	1.87						6.40	
	4	1.97						6	
Treated with 3% Solution	1	1.73	3.40	96.53	88.90	0.050	0.049	7.20	5.70
	2	1.98	3.62	82.82		0.049		6.40	
	3	1.76	3.43	94.88		0.50		4.12	
	4	1.92	3.48	81.25		0.049		5	
Treated with 5% Solution	1	1.70	3.26	91.76	90.32	0.078	0.082	8.47	5.90
	2	1.73	3.32	91.90		0.079		5.35	
	3	1.95	3.85	97.43		0.095		6.50	
	4	1.92	3.46	80.20		0.077		3.25	
Treated with 8% Solution	1	1.91	3.45	80.62	96.13	0.121	0.141	5.5	5.97
	2	2.01	4.13	105.47		0.169		3.62	
	3	1.85	3.66	97.83		0.144		8.12	
	4	1.65	3.31	100.60		0.132		6.64	

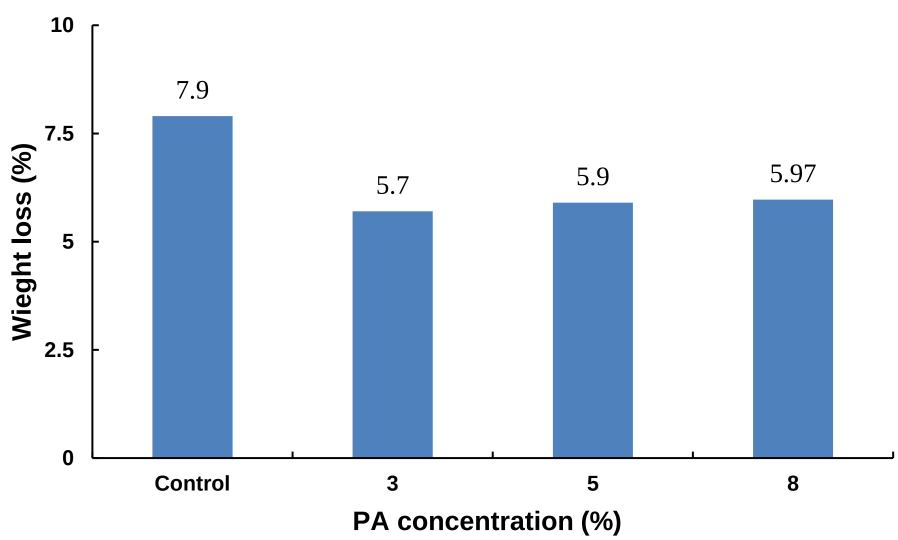

Fig. 5. Average weight loss of beech specimens treated with date palm waste derived PA: contaminated with *Trametes versicolor*

The average weight reduction based on weight after impregnation for the control set was 7.90%, and for the sets of treated samples with 3, 5, and 8% of solutions, the results were 5.70, 5.90, and 5.97%, respectively. These values are shown in Fig. 4. The weight reduction in treated samples, when compared with control ones, seemed remarkable.

Physical status of specimens after incubation period, is shown in Fig. 6. The 8% PA solution satisfactorily protected beech wood from *T. versicolor* (Fig. 6).

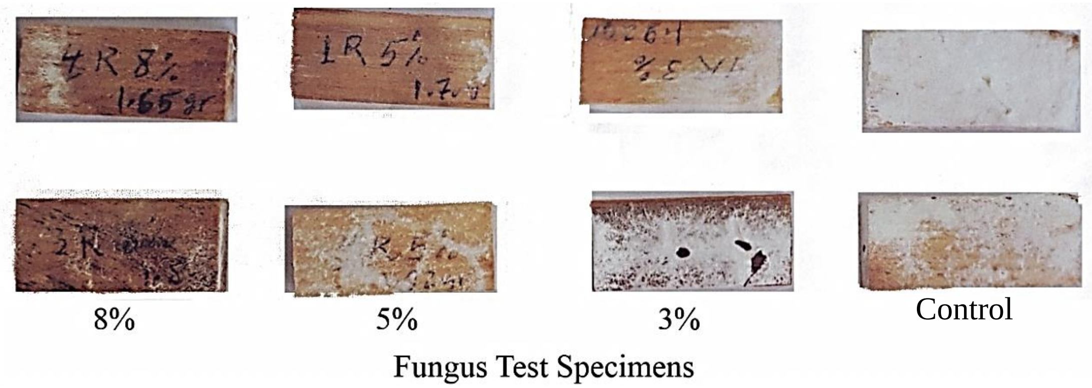

Fig. 6. Physical status of fungus contaminated samples after incubation: above row is pine sample, and below is poplar

Date palm wastes are fibrous materials and may seem to be an option in replacing and other natural fibers in producing fiber-based products. However, this does look right, since it will require continuous removal of wastes from palm groves, which in long run causes soil degradation in palm grooves, regarding its fertility and nutrient balance. In addition, collection and transportation of these wastes, a light and voluminous substance, to an installation producing fiber-based products with feasible capacity, may not be economically justified.

Productivity in utilization of date palm wastes by establishment of pyrolysis process installation is an approval approach, because it is less capital intensive and through this process two products are produced. Producing these two products with organic origin will be of no harm to the environment and their uses in agricultural activities are rather broad, including addition to the soils as fertilizer agent.

CONCLUSIONS

1. Results of testing pyrolysis-derived acid (PA) obtained from date palm wastes in controlling mold's growth on wood and consequent discoloration, have shown promise.
2. The effectiveness of this PA in terms of its concentration in treating solutions is species-dependent, because by visual inspection its 3% solution on pine wood performed better on poplar one. Therefore, effectiveness of its higher concentrations in treating solution may vary on different species of wood.
3. The application of 8% PA as a treating solution showed a successful result in protecting beech wood against *T. versicolor* fungus.
4. This PA with antifungal property can be used as an organic pesticide.

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