The Research of Optimal Conditions to Increase the Efficiency of Antipyren-Antiseptic Properties on the Basis of Processing of Sulfur Oligomers on Wood Materials

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Abstract:
The thermal stability of wood materials modified with oligomeric flame retardants and their optimal conditions have been determined by using electron microscopic analysis.

Key words: oligomer, flame retardant, termite, thermal stability, electron microscope, PO-1, PO-2, DGT-1, DGT-2 and DGT-3.

Thermal stability of wood materials treated with sulfur-containing compounds PO-1, PO-2, DGT-1, DGT-2 and DGT-3 brand flame retardants have been studied. Differential-thermogravimetric analysis of the synthesized briquettes has been performed on a derivatograph operating in the Paulik F, Paulik I, Erdey L.'s system. This method is based on the change of thermal effects of compounds in the temperature range of 293-793 K, when the temperature rise rate is 2-5 K / min.

The calculation of the effective kinetic parameters of the destruction of stabilized samples has been performed according to TGA data by using the Freeman and Carroll's method. The decomposition rate of the polymer is as follows:

dW/dt = (Ao/RH)e-E/RTWn (2.2)

where, RH-heating rate, W-polymer size, (the weight), A_o-predispositional multiplication, effect of *n*-reaction composition, effective activation energy of *E*-polymer thermodestruction. The order of the reaction *n* is found from the equation of the tangent angle of deviation of the graph of dependence in the logarithmic coordinates, and the effective activation energy of the thermodruction is determined from the intersection of the ordinate axis.

The weight loss kinetics of wood materials treated with PO-1 in relation to this process temperature are shown in Figure 1. Weight loss in the considered temperature range is associated with various processes: oxidation of the composite, decomposition with the release of volatile substances, and others. With the increase in temperature on the TGA curve, weight loss of wood materials treated with PO-1 brand flame retardant was observed at the destruction of the main part at temperatures from 200^{O}C to 300^{O}C. This composite lost 0.5mg weight at 120^{O}C and this figure was 12.6% of the total mass. The picture shows a weight loss of 2.19 mg at 300^{O}C, which is 53.1%. As a result of our

observation of thermal processes, an exothermic effect has also been detected with two heat emissions at the temperatures at which the main weight loss occurred at the temperatures of DTA 408.4^{O}C and 600 ^{O}C.

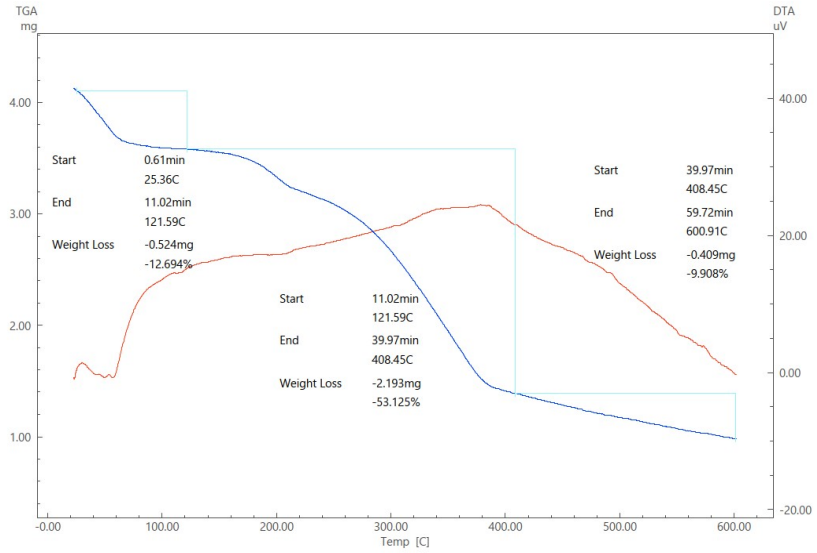


Figure 1. Thermal stability of wood materials treated with PO-1 brand flame retardant

The weight loss of wood materials treated with PO-2 brand flame retardant has comprised 0.28mg (9.6%) at 132^{O}C. Destruction of the main part was observed at 350^{O}C, which means 1.17 mg of weight loss and which is 39.3% of the total weight (50.0% of weight loss at 350^{O}C). At 428 ^{O}C, it has been 0.9 mg (30% of total weight). At a temperature of 600 ^{O}C, 0.62 mg of weight is lost, which is 21% of the total weight. As a result of our observation of thermal processes, an exothermic effect has also been detected, and at the temperatures of DTA 356.4^{O}C 382^{O}C, 400^{O}C and 480^{O}C, the condition with four heat outputs has been researched.

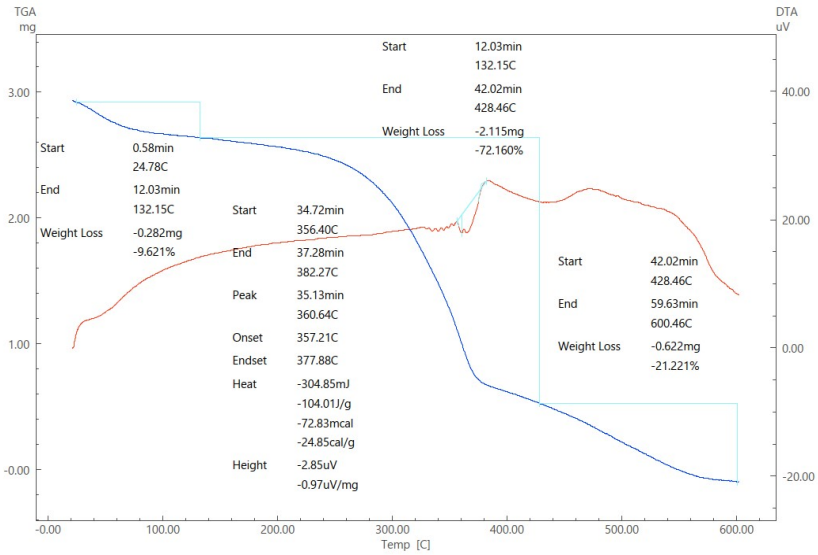


Figure 2. Thermal stability of wood materials treated with PO-2 brand flame retardant

The weight loss of wood materials treated with DGT-1 brand flame retardant has been 0.2mg (9.0%) at 100-132^{O}C. Destruction of the main part has been observed at the temperature of 350-393^{O}C

which means 1.8 mg of weight loss has been studied, it means 70% of the total weight (50.0% of the weight loss at 320^{O}C). At the temperature of 393-600^{O}C, it has been 0.5 mg (23% of the total weight). As a result of our observation of thermal processes, an exothermic effect has also been detected, and at the temperatures of DTA 320.4^{O}C, 380^{O}C, and 480^{O}C, the condition with three heat outputs has been researched.

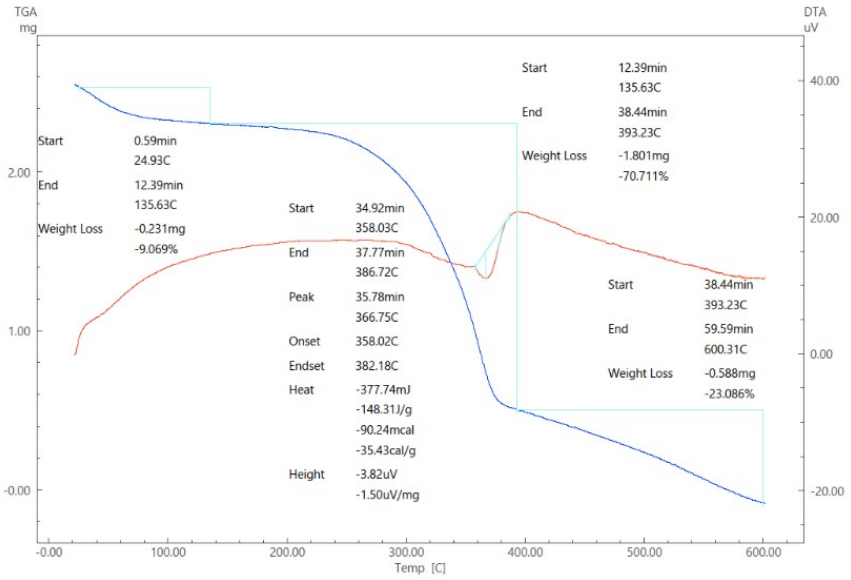


Figure 3. Thermal stability of wood materials treated with DGT-1 brand flame retardant

The weight loss of wood materials treated with DGT-2 brand flame retardant was 0.2mg (9.0%) at 100-132^{O}C. Destruction of the main part has been monitored at the temperature of 350-393^{O}C, which means 1.8 mg of weight loss has been studied and which is in turn accounted for 70% of the total weight (50.0% of the weight loss at 350^{O}C). At the temperature of 393-600^{O}C it has been 0.5 mg (23% of the total weight). As a result of our observation of thermal processes, an exothermic effect has also been detected, and it has been as researched with three heat outputs at the temperatures of DTA 320.4^{O}C, 380^{O}C and 480^{O}C.

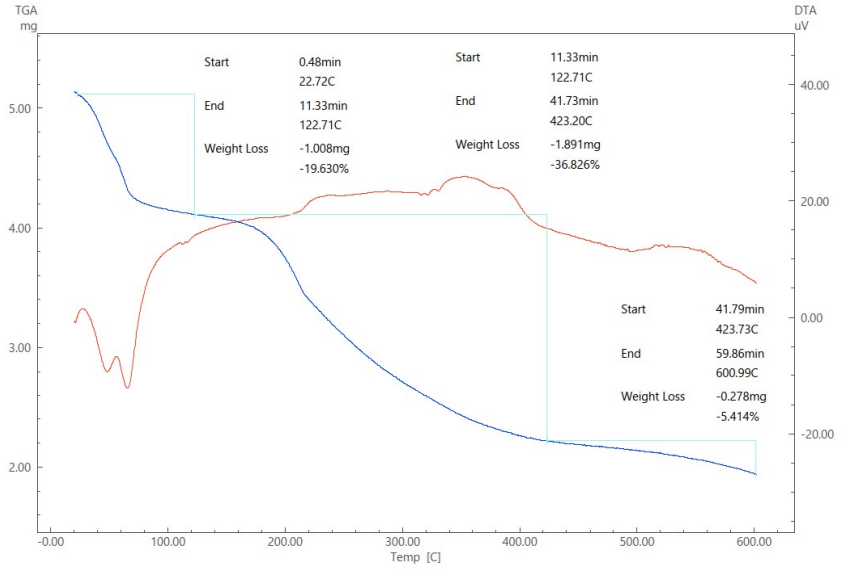


Figure 3. Thermal stability of wood materials treated with DGT-2 brand flame retardant

The weight loss of wood materials treated with DGT-3 brand flame retardant has been 1.7mg (25.2%) at 100-137^{O}C. Destruction of the main part has been observed at the temperature of 137-310oS which means 1.46 mg of weight lossand which is 21% of the total weight (50.0% of the weight loss at 420^{O}C). At the temperature of 310-600^{O}C, it has been 0.85 mg (12.6% of the total weight). As a result of our observation of thermal processes, exothermic effects have also been identified, and it has been studied with three heat outputs at the temperature of DTA 327.4^{O}C, 340^{O}C, and 490^{O}C.

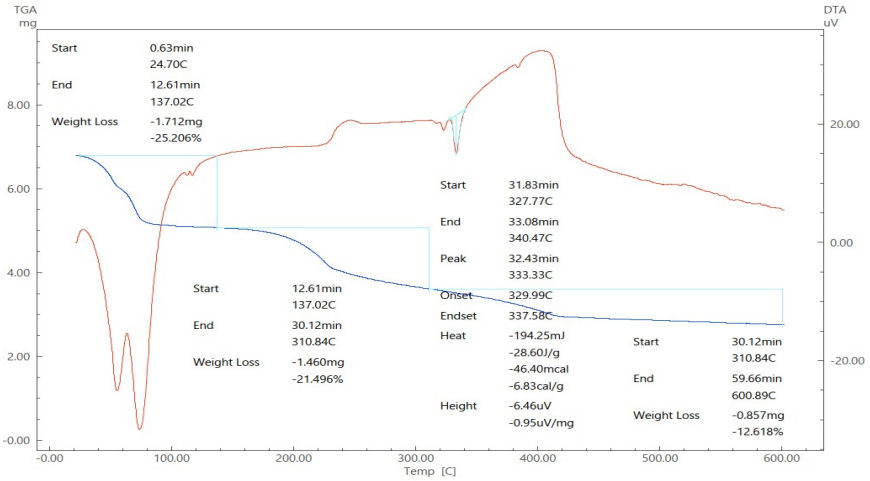


Figure 3. Thermal stability of wood materials treated with DGT-3 brand flame retardant

Thus, the study found that the mass loss of untreated wood materials in the temperature range of 130-350°C has been 67.8%, high thermal separation (exoffect) has been observed at 278°C, and the weight loss has been 50.2%.

The thermal stability of wood materials treated with PO-1, PO-2, DGT-1, DGT-2 and DGT-3 flame retardant compounds was 50% at a temperature of 310-420^{O}C and has been found to be higher than the thermal stability of untreated wood materials. The experimental test results have showed that the relatively high thermal stability of these PO-1, PO-2, DGT-1, DGT-2 and DGT-3 composites are DGT-2 and DGT-3 compounds.

Electronic mocroscope (SEM). Optical microscopic analysis to study the surface morphology of the composite has a great deal of importance in the study of wood building materials modified with sulfur-containing oligomeric flame retardants using the method of electronic microscopy. The study of the morphology of the hydrate phase and the change in the structure of the composition has been carried out by using an electron microscope of Jeol Interactive Corporation, Japan JSM-6460LA which has the following technical characteristics:

Fixed: 4.0 nm (at 30 Kv); Voltage acceleration: 0.1 to 4.9 Kv (with a step voltage of 10 V), with a step voltage of 5 to 30 Kv (100 V); Increase: from x8 to x300,000;

Electronization of the samples under an electron microscope for testing has been performed by vacuum dusting of a platinum layer 10-20 nm thick.

The sources in the literature and microscopy data from an X-ray microanalyzer from Oxford have been used for analyzing the images which provide 0.5% accuracy of the composition of the element phase.

By this method, it is possible to partially determine the composition of the processed wood sample by the absorption of antipyretic-antiseptics into the parts and their placement in the structure.

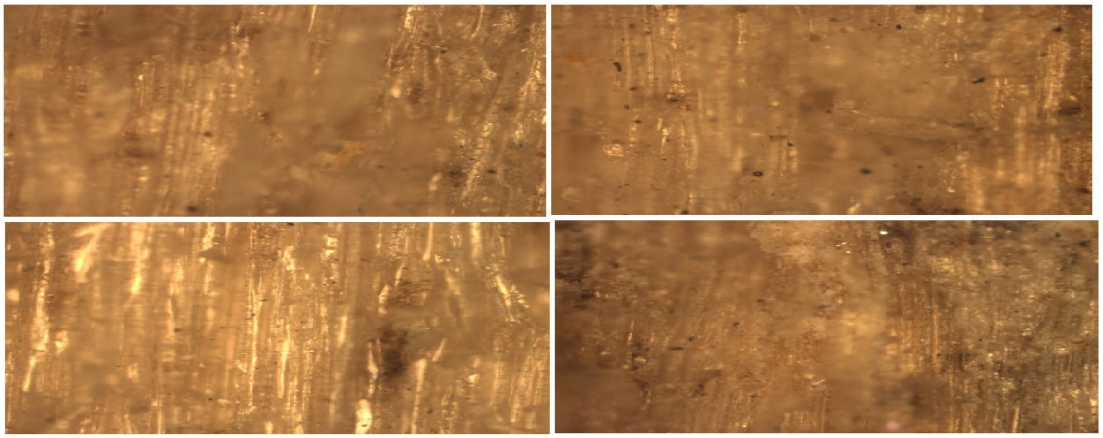


Figure 3.6. Electron microscopic analysis of (pine) wood materials untreated with flame retardants

Sulfur-containing oligomeric flame retardant antiseptics PO-1, PO-2,DGT-1, DGT-2 and DGT-3 have been analyzed by electronic microscopy of composites formed with wood materials. It can be seen that the sample shown in Figure 3.6 is a raw wood sample, and its structure is clearly visible. The study of recycled wood materials used using the optical microscopy method has showed that the structure is distinguished by the location of the particles in it, which indicates their high surface activity. (figures 3.7; 3,8; 3,9; 3,10).

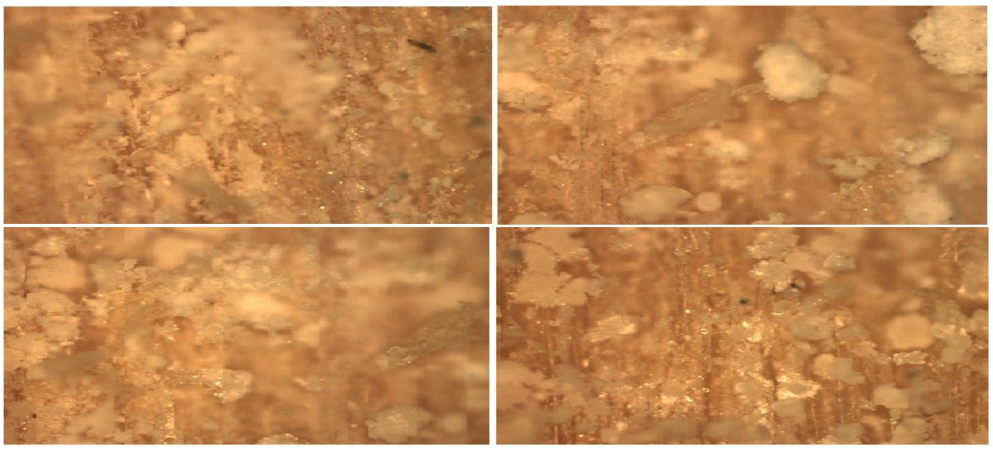


Figure 3.7. Electronic microscopic analysis of wood materials treated with PO-1 brand flame retardant antiseptics

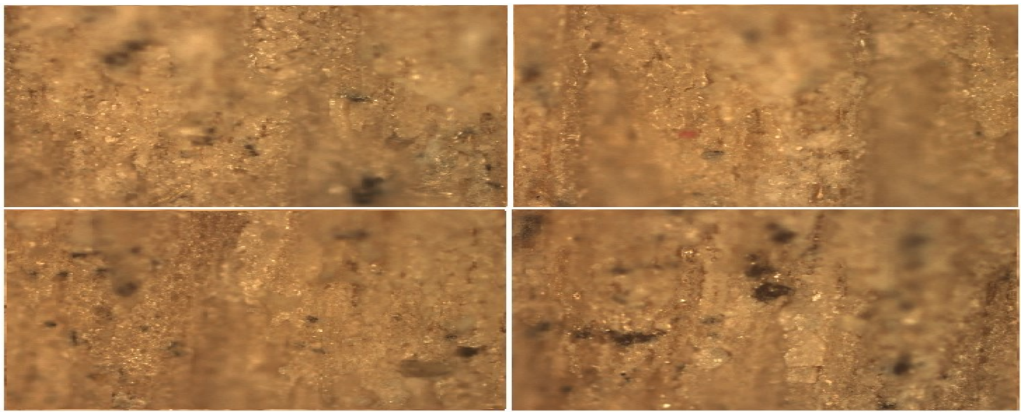


Figure 3.8. Electronic microscopic analysis of wood materials treated with PO-2 brand flame

retardant antiseptics

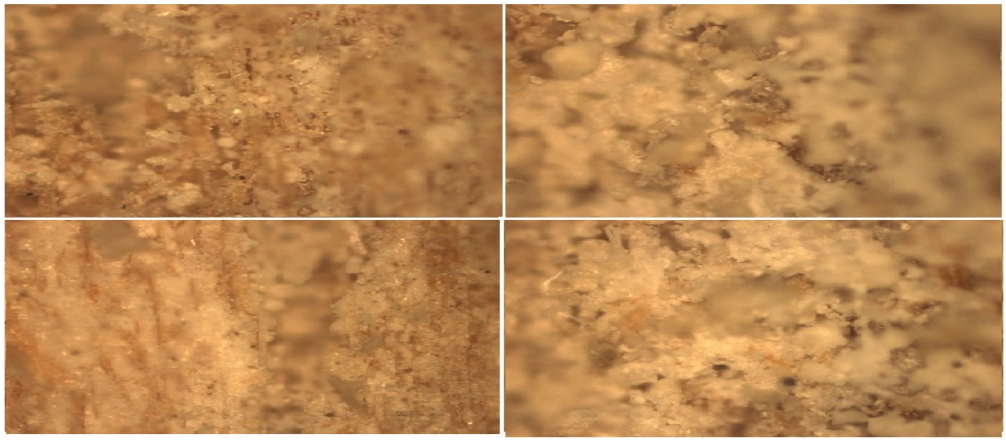


Figure 3.9. Electronic microscopic analysis of wood materials treated with DGT-1 brand flame retardants

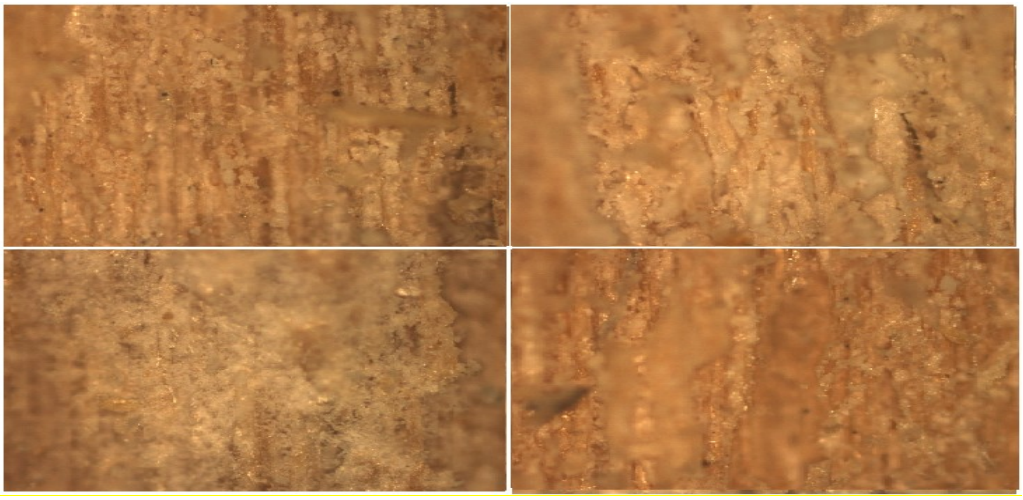


Figure 3.10. Electronic microscopic analysis of wood materials treated with DGT-3 brand flame retardants

In this analysis performed by using an optical microscope, the raw wood sample in Figure 3.6, and in figure 3.7; 3.8; 3.9; 3.10 it can be seen that the composition of the processed wood sample is evenly spaced in the wood sample cells. Based on the analysis of the results, the mechanism of interaction of flame retardant antiseptics with wood materials, consisting of sulfur, phosphorus, nitrogen, continues step by step: first, during the polymerization process, a liquid phase sol (colloidal state) is formed, which chemically binds to the active centers of the fiber and presents high antipyretic-antiseptic properties.

Many species have also been identified in Australia. White ants do not like cold weather which is why such weather is almost non-existent in countries. Termites cause countries to lose billions of dollars a year. This insect is also dangerous for cultural treasures as it may eats away even historical monuments.

There are only two types of termites in Uzbekistan: Turkestan termite and Caspian termite. By the middle of the twentieth century, the threat of termites had increased dramatically due to the influx of water to arid lands and the rise of groundwater. They escaped the water and came to the surface, finding the food they needed in the settlements. Another reason for their increase is the development

of desert areas, the use of timber nests by the population in the construction of buildings.

The researchers of the Tashkent Research Institute of Chemical Technology and Termez State University worked together to defeat these termites. As a result of the research, fire-resistant and termite-resistant composites have been created by obtaining sulfur-containing oligomeric flame retardants and processing them into wood building materials. In determining the biological effectiveness of wood materials treated with RO-1, RO-2, DGT-1, DGT-2 and DGT-3 sulfur-containing oligomeric flame retardant antiseptics, a pre-existing wood sample has been prepared in accordance with GOST 9.048-89 and tested. In table 2 shows the examination of the biological efficiency, chemical and corrosion resistance, effective application rate and application temperatures of the treated wood sample.

Table 2: Technical properties characterizing the biological efficiency of wood building materials

Samples	Biological efficiency GOST 9.048-89	Effective amount of application	Limited temperature of processing
Wood+PO-1	Point(score) 1	100-200 g/sq.m.	+40°C - 0°C
Wood +PO-2	Point(score) 1	100-200 g/sq.m.	+40°C - 0°C
Wood +DGT-1	Point(score) 1	100-150 g/sq.m.	+40°C - 0°C
Wood +DGT-2	Point(score) 1	100-200 g/sq.m.	+40°C - 15°C
Wood +DGT-3	Point(score) 1	1000 g/sq.m.	+40°C - +5°C

According to the results of the study, wooden building materials and structures treated with sulfur-containing oligomeric flame retardants can compete not only with biologically effective, environmentally and economically efficient analogues of the brand “Perilax” which are studied in analogues.

In our next experiments, the researches have been carried out in collaboration with researchers of “Khorezm Mamun Academy” to determine the biological effectiveness of PO-1, RO-2, DGT-1, DGT-2 and DGT-3 sulfur-containing wood materials treated with oligomeric flame retardant antiseptics, and the data on the predominant species of xylophagous insects *Hylotrupes bajulus, Anacanthotermes ahngerianus, Xylocopa valga, Anobium pertinax, A. turkestanicus* in anthropogenically transformed areas have been obtained, and a positive result of 84-90% has been achieved by increasing the biological efficiency of wood building materials using our proposed *“PO-1, PO-2, DGT-1, DGT-2 and DGT-3 brand oligomeric antipyretic antiseptics”* against them.

In addition, 10-20% aqueous and alcoholic solutions of oligomeric PO-1, PO-2, DGT-1, DGT-2 and DGT-3 flame retardants have been prepared, and $1m^2$ / 2.0 liter of solution has been soaked in a cellulose-rich sunflower stalk which is thought to be a termite feed, and it has been applied to the termites as food in a dried condition. During the experiment, the containers with special solutions in the laboratory was blurred, 25-30 working termites was used for each return, in general, the termites were given a specially prepared feed of 50 g.

Table 3: Biological efficiency of wood samples treated with PO-1oligomeric flame retardant antiseptics in the laboratory against xylophage-insects (in the example of termites)

Sample concentration, %	Number of dead termites in days (%)						Common number of termites	Biological efficiency (%)
	3 days	5 days	10 days	15 days	20 days	25 days		
RO-1 / 10	32±2	52±3	64±3	68±4	76±4	80±4	25,0±1,0	80±4

RO-1 / 15	40±1	56±2	72±3	76±3	84±3	88±2	25,0±1,0	88±2
RO-1 / 20	40±1	56±2	72±3	76±3	84±2	88±2	25,0±1,0	88±2
Control	-	-	-	-	-	-	-	-

Description: *(accuracy with respect to control n=5, M±m:P<0,01: <0,005)*

Besides, the specially prepared sunflower stalks were constantly moistened to maintain their softness. In the experimental test, termites were fed separately with filter paper and sunflower stalks that are not treated with flame retardants. According to experimental test results, most of the termites died within 25 days. For this time, controlled termites have found to be active in feed that were not been treated with flame retardants.(tables 3; 4; 5; 6; 7).

When PO-1 brand oligomeric flame retardants were applied as a feed to sunflower stalks soaked in 10-20% aqueous solution, the average number of dead termites in 3-10 days (10% solution) was increased by 32% - 64% and (15% -20% solution) 40-72%, then in 15-25 days (10% solution) it was 68% - 80%, and (15-20% solutions) 76-88%.

Table 4: Biological efficiency of wood samples treated with PO-2 oligomeric flame retardant antiseptics in the laboratory against xylophagous insects (in the example of termites)

Sample concentration, %.	Number of dead termites in days (%)						Total number of termites	Biological efficiency (%)
	3 days	5 days	10 days	15 days	20 days	25 days		
PO-2 / 10	32±2	48±3	56±4	64±3	68±4	76±5	25,0±1,0	76±5
PO-2 / 15	32±1	52±3	68±3	72±3	76±3	84±4	25,0±1,0	84±3
PO-2 / 20	40±1	56±2	72±3	76±3	80±2	84±2	25,0±1,0	84±2
Control	-	-	-	-	-	-	-	-

Description: *(accuracy with respect to control n=5, M±m:P<0,01: <0,005)*

When PO-2 brand oligomeric flame retardant antiseptic was applied as a feed to sunflower stalks soaked in 10-20% aqueous solution, the average number of dead termites in 3-10 days (10% solution) was 32%-56% and (15%-20% solution) 32- 78%, then in 15-25 days (10% solution) 64-76%, (15-20% solutions) 76-84%.

Table 5: Biological efficiency of wood samples treated with DGT-1 oligomeric flame retardant antiseptics in the laboratory against xylophagous insects (in the example of termites)

Sample concentration, %.	Number of dead termites in days (%)						Total number of termites	Biological efficiency (%)
	3 days	5 days	10 days	15 days	20 days	25 days		
DGT-1 /5	40±2	53±3	60±3	73±3	83±4	87±3	30,0±1,0	87±3
DGT-1/ 10	40±1	53±3	67±3	80±2	87±2	90±3	30,0±1,0	90±3
Control	-	-	-	-	-	-	-	-

Description: *(accuracy with respect to control n=5, M±m:P<0,01: <0,005)*

When DGT-1 oligomeric flame retardant antiseptics was applied as a feed to sunflower stalks soaked

in 5-10% alcohol solution, the number of termites that died in 3-10 days increased on average (5% solution) by 40% - 60%, (10% solution) by 40-67% , then on days 15-25 (5% solution) 73% - 87%, (10% solution) 80-90%.

Table 6: Biological efficiency of wood samples treated with DGT-2 oligomeric flame retardant antiseptics in the laboratory against xylophagous insects (in the example of termites)

Sample concentration, %.	Number of dead termites in days (%)						Total number of termites	Biological efficiency (%)
	3 days	5 days	10 days	15 days	20 days	25 days		
DGT-2/ 10	38±2	44±3	58±3	68±3	78±3	80±3	30,0±1,0	80±3
DGT-2/ 15	42±1	56±3	68±3	76±3	80±3	84±4	30,0±1,0	84±3
DGT-2/ 20	42±1	56±2	72±3	76±3	84±2	88±2	30,0±1,0	88±2
Control	-	-	-	-	-	-	-	-

Description: *(accuracy with respect to control n=5, M±m:P<0,01: <0,005)*

When sunflower stalks soaked in 10-20% aqueous solution of oligomeric flame retardant antiseptics DGT-2 were used as feed for termites, the average number of dead termites in 3-10 days (10% solution) increased by 38-58%, (15-20% solution) 42-72%, then in 15-25 days it has been (10% solution) 68- 80%, (15-20% solutions) 76-88%.

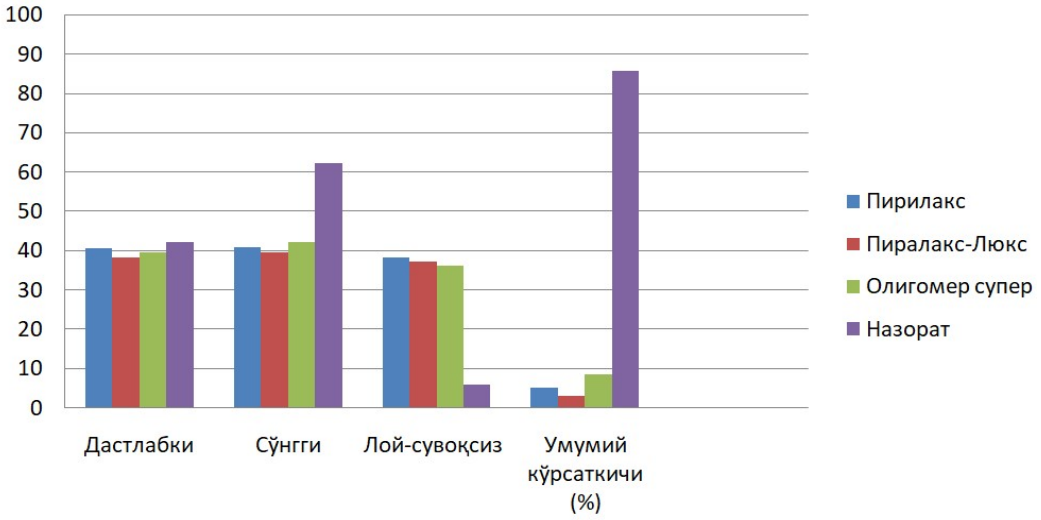
Table 7: Biological efficacy wood samples treated with DGT-3 oligomeric flame retardant antiseptics in the laboratory against xylophagous insects (in the example of termites)

Sample concentration, %.	Number of dead termites in days (%)						Total number of termites	Biological efficiency (%)
	3 days	5 days	10 days	15 days	20 days	25 days		
DGT-3/ K	10±1	10±3	17±4	20±3	23±3	23±3	30,0±1,0	23±3
Control	-	-	-	-	-	-	-	-

Description: *(accuracy with respect to control n=5, M±m:P<0,01: <0,005)*

Sunflower stalks have been coated and moistened with composite mixtures based on DGT-3 oligomeric flame retardant antiseptics, resulting in an average of 10-17% of dead termites in 3-10 days and 20-23% in 15-25 days when applied to termites as food. One of the main reasons for this is that this coating is partially absorbed into the interior of the wood stem and hardens in the solid state. This experiment caused the termites to pass through the coating by being wetted with water like other samples in the test work. However, in all cases it has been observed that the feeding process of the pest continues.

In the subsequent experimental tests, these processes are carried out in the natural environment of *Anacanthotermes* generation termites, and the imported analogues of *Pirilaks and Piralaks-Lux* antipyrene-antiseptic solutions, wood-based composites and local raw materials *PO-1, PO-2, DGT-1, DGT-2 and DGT-3* brand oligomer antipyrene-antiseptic solutions have been studied for attractiveness to the feeds based on composites processed into wood materials. (Figure 8).



(Initial;Last; Without Plasterwork;Common index%) (Pirilax, Pirilax-Luxe, Oligomer, Conrol)

Figure 8. The research has been carried out on the attractiveness of *Anacanthotermes* generation of termites to composites which are based on wood materials treated with antipyretic-antiseptic solutions

Conclusion. In the research of attractiveness of termites belonging to *Anacanthotermes* generation to the feeds treated with *PO-1, PO-2, DGT-1, DGT-2 and DGT-3* oligomeric flame retardant antiseptics under natural conditions, the size of the initial, final and feed cuts has been taken into account. Based on the overall result analysis, it has been found that the feeding of termites in the feed unit is affected by 5.2% - 8.6% and the antiseptics we offer is by 3.1-4.8%. It should be noted that DGT-3 coatings have the best performance in the natural environment without exposure to moisture.

According to the results of this experiment, it is necessary to take into account the expediency of continuous implementation of preventive measures to achieve chemical and biological effectiveness in the protection of xylophagous insects and the construction of durable, building materials.

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