THERMAL CONDUCTIVITY OF OAK IMPREGNATED WITH SOME CHEMICALS AND FINISHED

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The objective of this research was to investigate the effects of some impregnation materials and varnishes on the thermal conductivity of oak wood. Ammonium sulfate, borax, boric acid, zinc chloride, diammonium phosphate, and sodium silicate as impregnation chemicals and polyurethane, cellulosic, synthetic, coloured varnishes and cellulosic, synthetic, industrial paints as finishes were used. The wood materials were impregnated by using the vacuum-pressure method. The thermal conductivity test was performed based on the ASTM C 1113-99 hot-wire method. Results showed that the impregnation chemicals increased the thermal conductivity. The highest values were obtained with boric acid and sodium silicate. In addition, the thermal conductivity of painted oak was higher than that of varnished oak. The lowest thermal conductivity of 0.1465 Kcal/mh°C was obtained with the oak control. The highest thermal conductivity of 0.1756 Kcal/mh°C was obtained when oak was painted with industrial paint and impregnated with boric acid.

INTRODUCTION

Wood has been preferred as a residential construction material since ancient times because of its natural beauty and excellent properties, such as high specific strength, heat insulation, and ease of handling and processing (Kılıç et al. 2006). For example, wood's low thermal conductivity and good strength make it of special interest for building construction, refrigeration, automobile applications, and cooperage, among others (Gu and Sharp 2005).

Although wooden construction was more preferred in the past, reinforced-concrete constructions are more widely used currently. There are several reasons for this trend in Turkey. One of them is the traditional opinion that wood is not a suitable material from the point of view of fire safety. Flame retardant treated materials have much better fire performance than untreated wood products.

Thermal conductivity is a very important parameter in determining the heat transfer rate and is required for development of drying models and in industrial operations such as adhesive cure rate (Gu and Sharp 2005; Zhang and Datta 2004). Information on the thermal conductivity of wood and its relationship to other wood properties is of interest from the standpoint of thermal insulation, drying, plasticizing, preservation, gluing of wood, and where heat resistance of wood is a major consideration in its application (Sanyal et al. 1991; Hansson and Antti 2008).

Waterborne inorganic salts are a special group of penetrating finishes. These surface treatments result in a finish similar to semitransparent penetrating finishes because they change the color of the wood and leave a surface deposit of material similar to the pigment found in the semitransparent stains (Feist and Hon 1983).

Long-term retardants consist of the same inhibiting chemicals dissolved in water. They remain effective even after water has been removed by evaporation. The key ingredient in these retardants is the active retardant salt, usually referred to as "active salt," which is typically either an ammonium sulfate or ammonium phosphate (George and Johnson 1986). All salts may not be equally effective, when applied to wood in the same concentration. By adjusting the amount of salt applied to the wood, one may achieve the maximum performance.

Gratham et al. (1976) reported effects of exposure on the performance of different preservative varnish layers. In recent studies, one hundred and thirty individual chemicals or combinations of chemicals in the form of various salts have been evaluated for flame-spread reduction, smoke, and corrosivity (Levan 1984). Diammonium phosphate ranked first in reducing flame spread, followed by monoammonium phosphate, ammonium chloride, ammonium sulfate, borax, and zinc chloride. Zinc chloride, although excellent as a flame retardant, promoted smoke and glowing (Levan 1984).

Thermal conductivity is needed to conduct the heat from the surface to the inner parts during the combustion of wooden material. The lower the thermal conductivity, the more retarded the onset of combustion will be. Cell cavities of wooden material impregnated by fire retardant materials through the use of pressure and vacuum methods will be filled in with these chemicals. In this situation, as cell cavities of wooden material are filled, thermal conductivity increases compared to unimpregnated wooden material. On the other hand, because of the fire retardant characteristics, the burning of the material is either delayed or prevented.

Since there has not been sufficient study of the thermal conductivity of oak, the aim of this study was to compare the effects of impregnated materials (ammonium sulfate, borax, boric acid, diammonium phosphate, sodium silicate, zinc chloride) on the application and performance of finishing varnishes (polyurethane, cellulosic, synthetic) and paints (cellulosic, synthetic, industrial).

EXPERIMENTAL

Wood Materials

Oak (*Quercus petreae Mattu Liebl.)* was chosen randomly from timber suppliers of Ankara, Turkey. A special emphasis was put on the selection of the wood material. Accordingly, defect-free, whole, knotless, normally grown (without zone line, reaction wood, decay, insect damage, or fungal infection) wood materials were selected.

Impregnating Materials

Ammonium sulfate (AS), borax (BX), boric acid (BA), zinc chloride (ZC), diammonium phosphate (DP), and sodium silicate(SS) were used. These materials all have fire retardant character. Properties of the impregnating agents are shown in Table 1.

Table 1. Properties of Impregnation Chemicals Used

Impregnation Chemicals	Solution Conc. (%)	Solvent	Purity (%)	pH		Density (g/ml)	
				BI	AI	BI	AI
Ammonium sulfate	5	Pure water	97	6.5	6.5	1.06	1.07
Borax	2.5	Pure water	98	9.12	9.10	1.04	1.04
Boric acid	5	Pure water	98	5.23	5.30	1.02	1.02
Borax+Boric acid	5	Pure water	98	8.02	7.99	1.03	1.04
Zinc chloride	5	Pure water	99	5.91	5.72	1.07	1.07
Diammonium phosphate	5	Pure water	97	7.85	7.83	1.07	1.09
Sodium silicate	5	Pure water		11.16	11.1	0.92	0.95

BI:Before impregnation   AI: After impregnation

Varnishing and Painting Materials

Synthetic varnishes, polyurethane varnish, coloured varnish, or cellulosic varnish were used (Ozdemir 2003; Kurtoğlu 2000). In other cases, synthetic primer, industrial primer, or cellulosic primer were used (Disan Boya, Istanbul, Turkey 2008).

Determination of Density

Wood materials were kept in the room at $20 \pm 2°C$ and 65 ± 3 % relative humidity until their weight became stable (TS 2471, 1976). Air-dry densities of wood materials before and after impregnation used for the preparation of treatment samples were determined according to TS 2472 (1976). Afterwards, the dimensions of wood materials were measured by a compass of ±0.001 sensitivity and volumes were determined by a stereo-metric method. The air dry density (δ_{12}) was calculated by the following equation:

$$\delta_{12} = M_{12}/V_{12} \ \text{g/cm}^3 \tag{1}$$

where M_{12} is the air dry weight (g) and V_{12} is the volume (cm³) of the sample.

Preparation of Experimental Samples

Air-dry specimens with a dimension of 20 x 50 x 100 mm were cut from the drafts for impregnation. Then, wood samples cut from sapwood were kiln-dried at a temperature of 103 ± 2 °C. Then the wood samples were conditioned at 20 ± 2 °C and $65\pm 3\%$ relative humidity until they reached constant weight by holding them for 3 months in a climatization room. Before the impregnation process, the average moisture content of the samples was about 12%. A vacuum-pressure method was applied to the samples for impregnation, using a vacuum chamber of 31 cm diameter and 55 cm depth. The samples were weighed before impregnation and placed in a vacuum chamber. After the vacuum period (710 mm-Hg, 15 min), the impregnating solution containing ammonium sulfate, borax, boric acid, zinc chloride, diammonium phosphate, or sodium silicate was filled into the vacuum chamber until the samples were covered. Pressure was increased to 3 bar and maintained for 1 h. After the internal pressure was decreased to atmospheric

pressure, impregnation solution was unloaded and the final vacuum, 710 mm-Hg, was applied for 10 min. Then, the samples were removed and re-weighed. After the impregnation, all impregnated samples were held for 15 days in circulating air for evaporation of the solvent. After this period, the impregnated samples were kiln-dried at 103 ± 2°C until they reached constant weight. The dry weight of the samples was determined and recorded. The ratio of retention (R, %) were calculated as follows:

$$R(\%) = \frac{M_{di} - M_d}{M_d} \times 100 \qquad (2)$$

where M_{di} is the sample dry weight after impregnation (g), M_d is the sample dry weight before impregnation (g).

Finally the wood samples were conditioned at 20 ± 2°C and 65± 3% relative humidity until they reached constant weight by holding them for two weeks under normal conditions in a climatization room. After that, finish was applied according to principles of ASTM D-3024 (2008). The advice of the manufacturing factories was taken into consideration for the application of finish.

All processes were carried out at 20 ± 2°C. Finished test samples were kept at 20 ± 2°C and 65 ± 3% relative humidity until they reached constant weight.

Execution of the Test

A quick thermal conductivity measurement based on the ASTM C 1113-99 (2004) hot-wire method was used. The QTM 500 device is a product of Kyoto Electronics Manufacturing, Japan. A PD-11 box probe sensor (constantan heater wire and chromel alumel thermocouple) was used. A variable power supply (Variac) was used to supply constant electrical current to the resistance. The measurement range was 0.0116–6 W/mK. The measurement temperature was -100 to 1000 °C (external bath or electric furnace for temperature other than room). The sample size required was 20 x 50 x 100 mm. The measuring time, according to the standard, was 100 –120 s (Sengupta et al. 1992).

Data Analyses

By using seven finished materials and one control, seven impregnation chemicals and one control sample as parameters, a total of 320 samples (8x8x5) were prepared using 5 samples for each parameter. Multi-analyses of variance were used to determine the differences between the thermal conductivity of the prepared samples.

RESULTS AND DISCUSSION

The averages of density are given in Table 2. The highest density (0.652 g/cm³) was obtained for oak wood impregnated with boric acid. The lowest density (0.628 g/cm³) was obtained in untreated control samples.

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Table 2. Average Values of Density of Specimens (g/cm³)

Impregnation Chemicals	Air-dry density
Ammonium sulfate	0.650
Borax	0.635
Boric acid	0.652
Borax+Boric acid	0.640
Zinc chloride	0.642
Diammonium phosphate	0.645
Sodium silicate	0.647
Control	0.628

The highest retention amounts are shown in Table 3. The highest amount, 3.65%, was achieved following impregnation with boric acid. The lowest retention amount, equal to 1.06%, was after impregnation with borax.

Table 3. Amounts Retained by Specimens (%)

Impregnation Chemicals	Retention
Ammonium sulfate	3.56
Borax	1.06
Boric acid	3.65
Ba+Bx	1.94
Zinc chloride	2.24
Diammonium phosphate	2.76
Sodium silicate	2.97

The highest retention amounts of varnishes are given in Table 4. The highest value of 1.7 g was in the case of synthetic varnish (shine varnish) samples. The lowest varnish retention amounts, 0.5 g, were for cellulosic and polyurethane varnish samples.

Table 4. Finish Composition of Specimens (g)

Impregnate materials	Polyurethane Varnish	Cellulosic Varnish	Synthetic Varnish	Colored Varnish
Ammonium sulfate	0.7	0.6	0.9	0.8
Borax	1.3	1.2	1.7	1.5
Boric acid	0.5	0.5	0.8	0.8
Bx + Ba	0.9	0.7	1.1	0.9
Zinc chloride	1.1	0.9	1.4	1.3
Diammonium phosphate	0.9	0.6	1.0	0.9
Sodium silicate	0.8	0.7	1.0	0.9
Control	1.3	1.1	1.6	1.4

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The averages of thermal conductivity are given in Table 5. The highest thermal conductivity 0.1756 Kcal/mh°C was obtained in the case of finished industrial paint, impregnated with boric acid of samples. The lowest thermal conductivity 0.1465 Kcal/mh°C was obtained in the case of oak wood prepared, unprocessed impregnated and unprocessed surface (control).

Results of multi-variance analyses applied on the data obtained from the thermal conductivity test are given in Table 6. According to the variance analysis, the effects of impregnating material and the effects of finishing material were statistically significant. On the other hand, according to the variance analysis, the effects of impregnating materials and finishing material with each other were not statistically significant.

The Duncan test result is given in Table 7. The highest thermal conductivity of 0.1756 Kcal/mh°C was obtained with finished industrial paint, impregnated with boric acid. The lowest thermal conductivity of 0.1465 Kcal/mh°C was obtained in the case of untreated controls.

The Duncan test results for varnish are given in Table 7. The highest thermal conductivity of 0.1710 Kcal/mh°C was obtained with synthetic varnish on wood that had been treated with sodium silicate. The lowest thermal conductivity of 0.1562 Kcal/mh°C was obtained in untreated controls varnished polyurethane varnish.

The Duncan test results for paint are given in Table 7. The highest thermal conductivity of 0.1756 Kcal/mh°C was obtained in the case of finished industrial paint, impregnated with boric acid. The lowest thermal conductivity of 0.1579 Kcal/mh°C was obtained in control samples painted cellulosic paint.

The Duncan test for impregnation is given in Table 7. The highest thermal conductivity of 0.1756 Kcal/mh°C was obtained in impregnated with boric acid. The lowest thermal conductivity of 0.1495 Kcal/mh°C was obtained in the case of wood impregnated with borax. The specific effects of boric acid impregnation on wood thermal conductivity may be attributed to higher hygroscopicity and the higher density of samples, depending on the amount of retention. In this context, the different increases in thermal conductivity as a result of impregnation with the chemicals can be explained by considering the combination of the effects of density and hygroscopicity of samples and different thermal conductivities of these chemicals. First, the densities of impregnated samples were higher than those of control samples (Table 2). This result showed that the increase in densities of impregnated samples was caused mainly by deposition of impregnating chemicals in the wood. Şahin Kol and Altun (2009) reported that the thermal conductivity of wood is strongly dependent on density. Also, Bendtsen (1966) indicated that diammonium phosphate showed no significant change in equilibrium moisture content but borax, boric acid, zinc chloride and ammonium sulfate increased the moisture content of treated wood. Because the chemicals used take up water, the equilibrium moisture content of the impregnated samples was higher than that of control samples. Thermal conductivity of wood increases in a linear fashion with increase in moisture content. As moisture in wood increases, the space between the molecules and the amount of moisture molecules increase, which gives greater molecular mobility and results in greater transfer of energy (Gu 2001).

Table 5. Average Values of Thermal Conductivity Coefficients (Kcal/mh°C).

I. M.	Finished Mat.	Mean	St. dev.	I.M.	Finished Mat.	Mean	St. dev.
Ammonium sulfate	PolyV	0.1664	0.0022	Zinc Chloride	PolyV	0.1648	0.0011
	Cellulosic V	0.1665	0.0015		Cellulosic V	0.1650	0.0020
	Synthetic V	0.1708	0.0020		Synthetic V	0.1681	0.0011
	Color V	0.1692	0.0020		Color V	0.1679	0.0015
	Cellulosic P	0.1684	0.0033		Cellulosic P	0.1671	0.0022
	Industrial P	0.1736	0.0014		Industrial P	0.1721	0.0021
	Synthetic P	0.1689	0.0030		Synthetic P	0.1679	0.0019
	Control	0.1563	0.0018		Control	0.1528	0.0013
Borax	PolyV	0.1604	0.0020	Diammonium Fosfate	PolyV	0.1645	0.0015
	Cellulosic V	0.1612	0.0040		Cellulosic V	0.1651	0.0020
	Synthetic V	0.1647	0.0020		Synthetic V	0.1689	0.0019
	Color V	0.1626	0.0026		Color V	0.1681	0.0011
	Cellulosic P	0.1634	0.0034		Cellulosic P	0.1662	0.0012
	Industrial P	0.1674	0.0024		Industrial P	0.1719	0.0019
	Synthetic P	0.1649	0.0019		Synthetic P	0.1671	0.0021
	Control	0.1495	0.0015		Control	0.1534	0.0010
Boric Acid	PolyV	0.1667	0.0017	Sodium Silicate	PolyV	0.1681	0.0020
	Cellulosic V	0.1671	0.0021		Cellulosic V	0.1686	0.0010
	Synthetic V	0.1700	0.0020		Synthetic V	0.1710	0.0015
	Color V	0.1689	0.0019		Color V	0.1696	0.0026
	Cellulosic P	0.1690	0.0045		Cellulosic P	0.1690	0.0030
	Industrial P	0.1756	0.0030		Industrial P	0.1725	0.0025
	Synthetic P	0.1705	0.0020		Synthetic P	0.1701	0.0021
	Control	0.1570	0.0020		Control	0.1555	0.0015
Ba+Bx	PolyV	0.1621	0.0020	Control	PolyV	0.1562	0.0010
	Cellulosic V	0.1614	0.0014		Cellulosic V	0.1573	0.0020
	Synthetic V	0.1657	0.0030		Synthetic V	0.1606	0.0010
	Color V	0.1634	0.0004		Color V	0.1592	0.0020
	Cellulosic P	0.1644	0.0020		Cellulosic P	0.1579	0.0030
	Industrial P	0.1682	0.0012		Industrial P	0.1623	0.0023
	Synthetic P	0.1654	0.0024		Synthetic P	0.1581	0.0019
	Control	0.1514	0.0014		Control	0.1465	0.0015

I.M. Impregnated Materials, V; Varnish P; Paint

Table 6. Multiple Variance Analysis for the Effect of Impregnating Materials, and Surface on Thermal Conductivity

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1.566E-02	63	2.486E-04	26.298	0.000
Intercept	8.780	1	8.780	928940.150	0.000
Factor A	7.722E-03	7	1.103E-03	116.706	0.000
Factor B	7.526E-03	7	1.075E-03	113.748	0.000
A*B	4.121E-04	49	8.410E-06	0.890	0.681
Error	2.420E-03	256	9.452E-06		
Total	8.799	320			
Corrected	1.808E-02	319			R: 0.866

Factor A = Impregnate materials (AS, BX, BA, BX+BA, ZC, DP and SS)
Factor B = Finished (varnishes (polyurethane, cellulosic, synthetic) and paints (cellulosic, synthetic, industrial)

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Table 7. Duncan test results of ımpregnate materials, varnishes and paints.

Factors	Samples	Mean	Homogeneous group	Factors	Mean	Homogeneous group
Unprocessed wood	5	0.1465	a	AS-Polyurethane V	0.1664	qprstuvwy
BX-C	5	0.1495	ab	AS-Cellulosic V	0.1665	prstuvwy
BA+BX-C	5	0.1514	bc	BA-Polyurethane V	0.1667	prstuvwyz
ZC-C	5	0.1528	bcd	BA-Cellulosic V	0.1671	rstuvwyzA
DP-C	5	0.1534	cde	ZC-Cellulosic P	0.1671	rstuvwyzA
SS-C	5	0.1555	def	DP-Synthetic P	0.1671	rstuvwyzA
C- Polyurethane V	5	0.1562	def	BX-Industrial P	0.1674	rstuvwyzA
AS-C	5	0.1563	def	ZC- Coloured V	0.1679	stuvwyzAB
BA-C	5	0.1570	efg	ZC- Synthetic P	0.1679	stuvwyzAB
C- Cellulosic V	5	0.1573	fgh	ZC- Synthetic V	0.1681	stuvwyzAB
C- Cellulosic P	5	0.1579	fghi	DP- Coloured V	0.1681	stuvwyzAB
C- Synthetic P	5	0.1581	fghi	SS- Polyurethane V	0.1681	stuvwyzAB
C- Coloured V	5	0.1592	fghij	BA+BX-Industrial P	0.1682	stuvwyzAB
BX- Polyurethane V	5	0.1604	ghijk	AS-Cellulosic P	0.1684	stuvwyzABC
C- Synthetic V	5	0.1606	ghijkl	SS-Cellulosic V	0.1686	stuvwyzABC
BX- Cellulosic V	5	0.1612	hijklm	AS- Synthetic P	0.1689	tuvwyzABC
BA+BX- Cellulosic V	5	0.1614	ijklmn	BA- Coloured V	0.1689	tuvwyzABC
BA+BX- Polyurethane V	5	0.1621	jklmno	DP-Synthetic V	0.1689	tuvwyzABC
C-Industrial P	5	0.1623	jklmnoq	BA-Cellulosic P	0.1690	tuvwyzABC
BX- Coloured V	5	0.1626	jklmnoqp	SS-Cellulosic P	0.1690	tuvwyzABC
BX- Cellulosic P	5	0.1634	klmnoqpr	AS- Coloured V	0.1692	uvwyzABC
BA+BX- Coloured V	5	0.1634	klmnoqpr	SS- Coloured V	0.1696	vwyzABCD
BA+BX- Cellulosic P	5	0.1644	klmnoqprs	BA- Synthetic V	0.1700	wyzABCD
DP- Polyurethane V	5	0.1645	lmnoqprs	SS- Synthetic P	0.1701	vwyzABCD
BX- Synthetic V	5	0.1647	mnoqprst	BA- Synthetic P	0.1705	yzABCD
ZC- Polyurethane V	5	0.1648	mnoqprst	AS- Synthetic V	0.1708	zABCD
BX- Synthetic P	5	0.1649	mnoqprstu	SS- Synthetic V	0.1710	ABCD
ZC- Cellulosic V	5	0.1650	mnoqprstu	DP-Industrial P	0.1719	BCDE
DP- Cellulosic V	5	0.1651	mnoqprstu	ZC-Industrial P	0.1721	BCDE
BA+BX- Synthetic P	5	0.1654	noqprstuv	SS-Industrial P	0.1725	CDE
BA+BX- Synthetic V	5	0.1657	oqprstuv	AS-Industrial P	0.1736	DE
DP- Cellulosic P	5	0.1662	oqprstuvw	BA-Industrial P	0.1756	E

AS; Ammonium sulfate, BX; Borax, BA: Boric acid, ZC; Zinc chloride, DP; Diammonium phosphate, SS; Sodium silicate, C; Control; V; Varnish P; Paint C; Control

According to Fig. 1, by using impregnating materials in preparation for varnish materials, the lowest thermal conductivity value was obtained in the case of control samples (unvarnished materials). Samples that were not impregnated gave the lowest values. Consequently, it can be inferred that the thermal conductivity values of wood materials that were impregnated were higher when compared to wood materials that were not exposed to the same process. Synthetic varnished samples give the highest values when compared to other varnished wood materials. The reason for this increase is that synthetic varnishes (shine) comprise more layers on a surface.
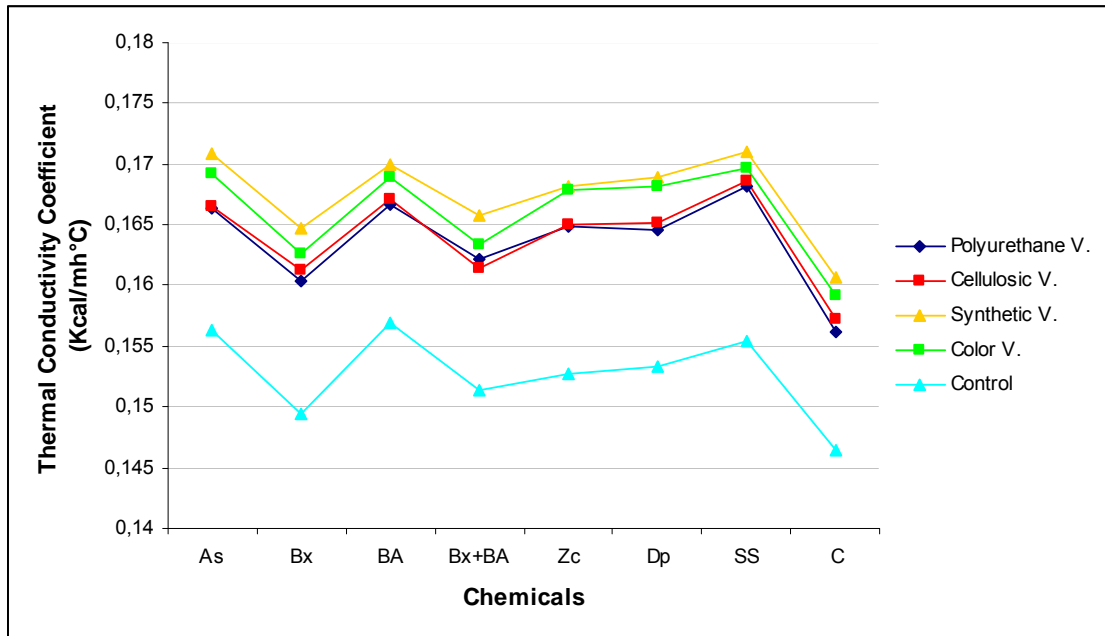
Figure1. Effect of impregnating materials and varnish materials on thermal conductivity

According to Fig. 2, the unpainted wood material that was lowest in thermal conductivity values was painted after impregnation. In unpainted control samples, boric acid gave the highest thermal conductivity values. Again, the reason for this is that the density of samples impregnated with boric acid is quite high depending on the amount of retention. Sample wood materials that were not impregnated gave the lowest values.
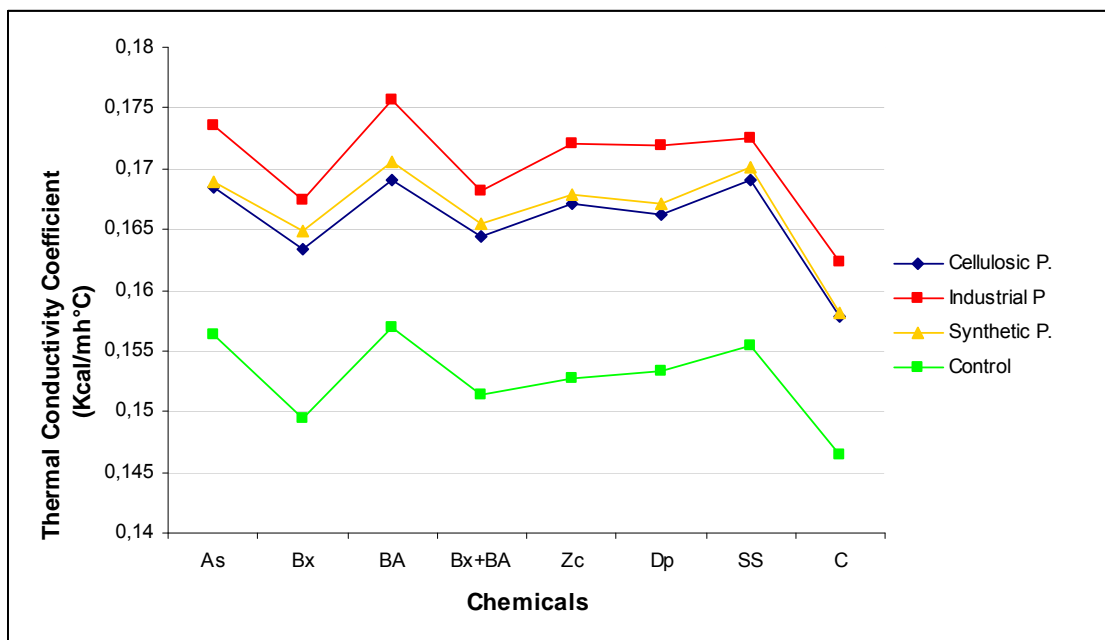
Figure 2. Effect of impregnating materials and paint materials on thermal conductivity

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CONCLUSIONS

1. When considering the thermal conductivity values of impregnated and varnished wood material, synthetic varnished samples gave the highest values when compared to other varnished wood materials. It was observed that while synthetic varnish increased conductivity values by 9.5% on average, colored varnish increased conductivity values by 8.5% on average, cellulosic varnish yielded increases of nearly 7.5%, and polyurethane varnish increased conductivity by 7% on average.

2. In the case of impregnated and painted wood samples, industrial paint samples gave the highest values when compared to other painted wood materials. It was observed that while industrial paint increased conductivity values by 11.5% on average, synthetic paint increased conductivity by nearly 9% and cellulosic paint increased it by 8.5% on average.

3. One of the most important observations here is that materials exhibiting lower amounts of retained material after impregnation gave higher thermal conductivity values, rather than the materials for which the amount of which retention was higher after the finished is completed.

5. Consequently, wood that was impregnated with boric acid, then finished with either synthetic varnish or industrial paint can be used in applications where thermal conductivity is required. Wood impregnated with borax, then finished with polyurethane varnish or cellulosic varnish can be used in applications where insulation is required.

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