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Effect of Adding Dibutyltin Dilaurate on the Quality of Polyurethane Paint for Applications in the Automotive Industry

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***Abstract*—In the automotive paint industry, the paint is attached by spraying, followed by a drying process using a baking oven process. For bumpers made from polypropylene (PP) plastic, the oven drying temperature is 80°C. The drying process is often difficult because it requires low temperatures and extended drying times. Therefore, additional raw materials (additives) are needed, which function as catalysts to speed up the drying process. Dibutyltin dilaurate (DBTDL) is an organotin catalyst used in the paint industry. This research aimed to investigate the effect of DBTDL addition on the properties of polyurethane paint used in the automotive industry. The variables of this research include concentration, temperature, and drying time. The properties tested were hardness, adhesion, and gloss. The research method consisted of three stages, namely the preparation stage, application stage, and testing stage. The research results found that the best hardness was obtained at temperatures of 80°C and 90°C, concentrations of 0.2% and 0.3%, and drying times of 25 minutes and 30 minutes. The adhesion test showed 100% results for all samples except at temperatures of 80°C and 90°C, a concentration of 0.3%, and a drying time of 30 minutes. The gloss test results showed a value of more than 90%. From the results of hardness, adhesion, and gloss tests, it can be concluded that the best condition was achieved at a concentration of 0.2% with a drying time of 30 minutes and a temperature of 80-90°C.**

***Keywords*— Automotive; catalyst; coating; dibutyltin dilaurate; polyurethane paint.**

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I. INTRODUCTION

Paint is used in various industries, including the automotive industry. There are several polymers for paint applications, one of which is polyurethane. Polyurethane paint is composed of two components. The first component contains hydroxyl groups (OH) present in the main component, while the second component contains isocyanate found in the hardener [1], [2]. These two components react to form cross-linking solid bonds [3]. Resin in paint contains alcohol groups (polyol), while the drying agent (hardener) contains polyisocyanate [4]. Thinner acts as a solvent to facilitate the paint application [5].

Polyurethane paint has low viscosity (1 Pa.s), low cross-linking temperatures (50 - 70°C), and fast gel time (less than 1 second) [6]. The resulting paint layer exhibits excellent gloss, weather resistance, solvent resistance, and a smooth texture [7]. Polyurethane paint's properties include strength and hardness, heat resistance, scratch resistance, flexibility, rapid surface coverage, and durability [3], [8].

In the automotive industry, several parts require painting, including the main part, namely the car body, which is made of steel, and the bumper, which is made of plastic [9]. A drying oven temperature of 140-150°C is used for the steel car body parts. An oven drying temperature of 140-150°C is usually used for car body parts made of steel. Meanwhile, for the bumper made of polypropylene (PP) plastic, an oven drying temperature of around 80-90°C is used [10], [11]. The drying process is often challenging because the temperature is not high and requires a long time [5]. Therefore, additional raw materials (additives) are needed to function as catalysts to speed the drying process [12].

Adding a catalyst to the paint mixture will speed up the drying time of the paint applied to the substrate surface without making a significant difference in its quality [12]. Dibutyltin dilaurate (DBTDL) catalyst is an organotin catalyst used in the paint industry [13]. This catalyst accelerates polymerization reactions in acrylic, polyester, and polyurethane resins [14]. DBTDL has highly reactive properties and is effective in increasing the speed of polymerization reaction, so it can produce paint products that dry faster and are more durable [15].

DBTDL offers several advantages over other catalysts. This catalyst demonstrates high activity with both aromatic and aliphatic isocyanates and high catalytic activity at deficient concentrations [16], [17]. Additionally, it boasts good solubility and compatibility with most common formulations. DBTDL also exhibits high tolerance to the influence of other materials in coating formulations, relatively low yellowing effects, and reasonably good stability against air, light, and moisture [16]. The excellent catalytic performance of DBTDL is based on its Lewis Acid properties [18]. Literature sources indicate that the complexation of tin centers in DBTL with NCO groups is one of the critical steps in urethane reaction catalysis [19].

Several studies have evaluated the use of DBTDL catalysts in the paint industry. One such study demonstrated that using DBTDL catalysts in the production of acrylic paint can increase the polymerization reaction rate and produce more durable paint products. Their study showed that using a DBTDL catalyst can reduce paint drying time and enhance the adhesion strength of the paint on applied surfaces [20]. Furthermore, another study showed increased hardness but decreased gloss value by adding DBTDL in acrylic paint [5].

The quality of painted products is crucial in the automotive industry. Understanding the factors influencing product defects facilitates improvements in processes, materials production, and evaluating tools or machines used in applications. Several specifications are considered in assessing the quality of paintwork [21]. Quality control of painted products is carried out through several testing methods, including visual inspection, gloss testing, hiding power testing, adhesion testing, hardness testing, and bending testing.

Visual inspection aims to detect defects in the paintwork, such as popping, pinholes, orange peel, cratering, mottling, sagging, dry spray, dirt, speckling, and other imperfections [22]. Gloss testing aims to measure the sheen of the paint film [23]. Hiding power testing measures the paint's coverage ability. The higher the hiding power, the thinner the paint film is required to cover the surface, and vice versa [21]. Adhesion testing measures the paint's bonding strength to the metal or plastic substrate to prevent peeling. This is typically done using the cross-cut method [24]. Hardness testing aims to determine the hardness level of the paint layer [25], [26]. The bending test assesses the paint's flexibility. It ensures that the coated object does not crack or peel off when subjected to bending or impact [21]. Fourier Transform Infrared Spectroscopy (FTIR testing is also necessary to observe any changes in paint compounds after adding additives [27].

The addition of a DBTDL catalyst is expected to accelerate the paint-drying process. It is also anticipated that this catalyst addition will not affect the quality of the paint, meeting the existing specifications and standards. The present study aims to investigate the effect of adding a DBTDL catalyst on the qualities of polyurethane paint. Previous studies have reported the addition of DBTDL catalyst in acrylic paint, however, to the best of our knowledge, no study has reported on the addition of DBTDL catalyst in polyurethane paint for automotive industry application.

II. MATERIALS AND METHOD

A. Dilution of DBTDL

The dilution process was carried out in the following steps. First, 10 ml of pure DBTDL was taken using a pipette, then poured into a 1000 ml measuring flask. The next step was to add the organic solvent xylene until the volume reaches 1000 ml. Then the flask was shaken until homogeneous and it was ensured that all the DBTDL was completely dissolved indicated by the disappearance of the yellow substance.

B. Sample preparation

The steps for sample preparation were as follows. First, 100 ml of polyurethane paint was put into a glass beaker. Next, the paint mixture was added to the DBTDL catalyst solution, which was diluted into varying concentrations, namely 0%, 0.1%, 0.2%, and 0.3% (from the total volume of the paint mixture). The mixture was then stirred using a mixer for 10 minutes at 600 rpm. Then, thinner and hardener were mixed with a ratio of paint to thinner of 1:1 and paint to hardener ratio of 4:1.

C. Paint application

The paint application was conducted as follows. Using a spray machine, the sample was applied to a 10 x 15 cm plate substrate. The size of the spray gun needle used was 1.5 mm, the wind pressure of 3.5 bar, the distance between the spray gun and the substrate was 10 cm, and the number of layers of paint to be applied was 2 layers. Then, the plate was left for a flash-off time (waiting time) of 5 minutes. After that, the plate was put into an oven, which had variable temperatures of 60°C, 70°C, 80°C, and 90 °C. Next, the plate was removed from the oven by varying drying times from 15 minutes, 20 minutes, 25 minutes, and 30 minutes.

D. Testing

Painting product quality testing carried out includes:

1) Adhesion: Coating adhesion testing aims to measure the level of paint adhesion to work objects, both metal and plastic. The purpose of this test is to prevent peeling on painted specimens. Testing was carried out using the cross-cut method by making 100 squares measuring 1 x 1 mm. The cut was made to the bottom of the paint layer on the specimen. The tool used in this test was Super Cutter Guide No. 315, Taiyu, Japan.

2) Hardness: Hardness testing aims to determine the paint layer's hardness level. The method was to use a pencil based on the level and scratch it onto the paint surface at a 45° angle [29], [30]. The tool used in this test was HI Uni Pencil, Mitsubishi, Japan. The pencil's hardness levels from the lowest to the highest are as follows: 2B → B → HB → F → H.

3) Gloss: Gloss-level measurements significantly influence an observer's first impression of the paint. With this method, the gloss on the applied paint film layer was measured at an angle of 60° [5]. The higher the gloss value, the better the shine. The tool used in this test was a Gloss checker type/model IG-320, Horiba, Japan.

III. Results and Discussion

A. Hardness

Table 1 shows that at a drying temperature of 60°C for all drying times, the increase in hardness level was observed when the DBTDL concentration was 0.2 and 0.3%. At 60°C, adding DBTDL at a low concentration (0.1%) did not affect the hardness level. Meanwhile, at higher drying temperatures (70°C-90°C), adding DBTDL increased the hardness level. It can also be observed that high hardness levels could be obtained without adding a DBTDL catalyst when the drying temperature was 90°C. This is because at high temperatures, drying proceeds faster, leading to increased hardness values. On the other hand, at lower drying temperatures, a higher amount of catalyst is needed to aid the polymerization process during drying. Table 1 presents the hardness test results at varying DBTDL concentrations, drying temperatures, and drying times.

TABLE I
HARDNESS TEST RESULTS

Concentration (%)	Temperature (°C)															
	60				70				80				90			
	Drying time (minutes)				Drying time (minutes)				Drying time (minutes)				Drying time (minutes)			
	15	20	25	30	15	20	25	30	15	20	25	30	15	20	25	30
0	2B	2B	2B	2B	2B	2B	2B	2B	B	B	HB	HB	B	HB	HB	HB
	2B	2B	2B	2B	2B	2B	B	B	B	B	B	B	B	HB	HB	HB
0.1	2B	2B	2B	2B	2B	B	B	B	B	HB	HB	HB	HB	HB	HB	HB
	2B	2B	2B	2B	B	B	B	HB	B	B	HB	HB	HB	HB	HB	HB
0.2	2B	B	B	B	B	B	HB	HB	HB	HB	HB	F	HB	HB	HB	F
	2B	2B	B	B	B	B	HB	HB	HB	HB	HB	HB	HB	HB	HB	HB
0.3	B	B	B	B	B	B	HB	HB	HB	HB	HB	F	HB	HB	F	F
	2B	B	B	B	B	B	HB	HB	HB	HB	HB	HB	HB	HB	HB	HB

Table 1 shows that the highest hardness level (F) could be achieved by adding a DBTDL catalyst at a concentration of 0.2% when drying at 80 °C for 30 minutes.

B. Adhesion

The adhesion test was conducted using the cross-cut method by making square cuts measuring 1 x 1 mm, totaling 100 squares. In this adhesion test, if any of the 100 squares peel off, the sample cannot be used as it does not meet the quality standards in the paint industry. Table 2 shows the results of the adhesion test. The results indicated that the addition of DBTDL catalyst at a concentration of 0.3% failed to meet the quality standard regarding paint adhesion when the drying process proceeded at 80°C and 90°C for 30 minutes. The poor adhesion ability occurred because, at high catalyst concentrations and high temperatures, the polymerization reaction becomes too fast.

TABLE II
ADHESION TEST RESULTS

Concentration (%)	Temperature (°C)															
	60				70				80				90			
	Drying time (minutes)				Drying time (minutes)				Drying time (minutes)				Drying time (minutes)			
	15	20	25	30	15	20	25	30	15	20	25	30	15	20	25	30
0	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
0.1	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
0.2	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
0.3	100	100	100	100	100	100	100	100	100	100	100	95	100	100	100	91
	100	100	100	100	100	100	100	100	100	100	100	93	100	100	100	88

An excessively rapid reaction may lead to uneven or imperfect polymer network formation, creating a brittle and less elastic paint film. As a result, the adhesion of paint to the substrate decreases. Accelerated polymerization at high temperatures and with a high amount of catalyst can cause the formation of unstable or non-uniform microstructures within the paint layer. These unstable microstructures can result in the paint layer's inability to bond to the substrate strongly, thus reducing its adhesion. Therefore, adding an appropriate amount of catalyst and using suitable drying temperatures to meet the paint's quality standard is necessary.

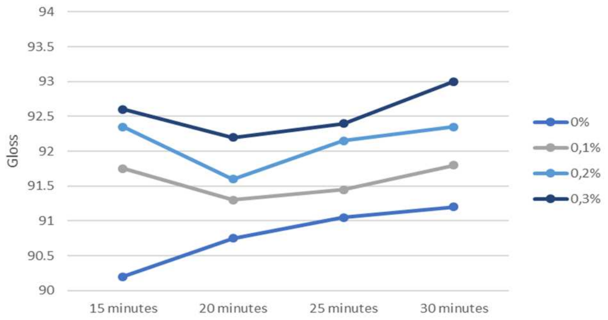
C. Gloss

The gloss test determines the shine of the samples applied to the substrate. This test utilizes the Gloss checker type/model IG-320. The research results using variations of DBTDL concentrations, drying temperatures, and drying times yielded the following gloss test results, as presented in Table 3. The quality standard requires good paint to have a gloss value of at least 80%. From Table 3, it can be seen that all samples meet the gloss standard.

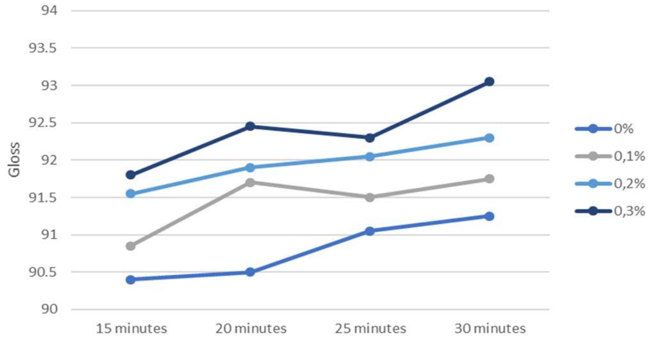
TABLE III
GLOSS TEST RESULTS

Concentration (%)	Temperature (°C)															
	60				70				80				90			
	Drying time (minutes)				Drying time (minutes)				Drying time (minutes)				Drying time (minutes)			
	15	20	25	30	15	20	25	30	15	20	25	30	15	20	25	30
0	90.1	90.8	91.1	91.2	90.5	90.5	91	91.2	90.7	90.6	91.5	91.6	90.5	91.3	91.7	91.3
	90.3	90.7	91.0	91.2	90.3	90.5	91.1	91.3	90.5	90.7	91.4	91.8	90.8	91.1	91.4	91.3
0.1	91.8	91.3	91.5	91.9	90.8	91.9	91.5	91.8	90.9	91.2	91.9	92.3	91.5	92.1	92	92.3
	91.7	91.3	91.4	91.7	90.9	91.7	91.5	91.7	91.1	91.2	92.7	92.4	91.4	92	92.2	92.4
0.2	92.4	91.5	92.1	92.3	91.5	91.9	92	92.2	92	91.9	92.7	92.8	92.4	92.8	92.3	92.7
	92.3	91.7	92.2	92.4	91.6	91.7	92.1	92.4	92	92	92.8	92.9	92.4	92.6	92.2	92.5
0.3	92.7	92.2	92.3	93	91.7	92.5	92.3	93	92.5	92.6	92.9	93.3	93	93.1	93.1	93.5
	92.5	92.2	92.5	93	91.9	92.4	92.3	93.1	92.5	92.7	92.9	93.2	92.8	93	93.1	93.3

Fig. 1 (a)-(d) shows the gloss values for different concentrations of DBTDL at drying temperatures of 60°C, 70°C, 80°C, and 90°C. From Fig. 3.1 it can be concluded that generally, the addition of DBTDL catalyst proved to increase the gloss value of the paint. Higher concentrations of DBTDL catalyst resulted in the higher values of gloss. Fig. 3.1 also shows that generally, the values of gloss increased with the increased in drying durations.



(a)



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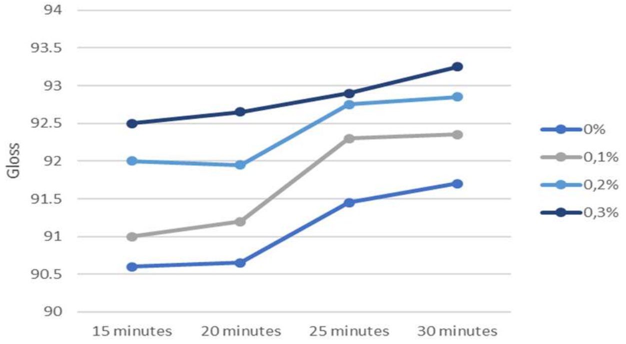
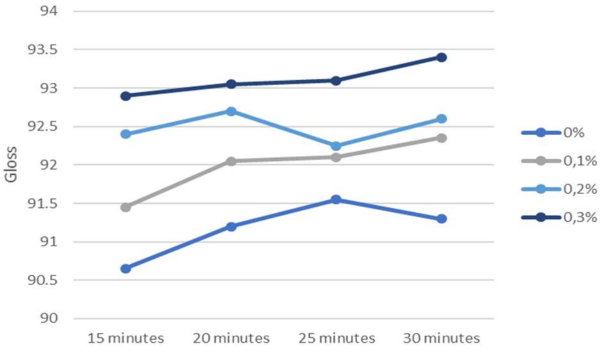


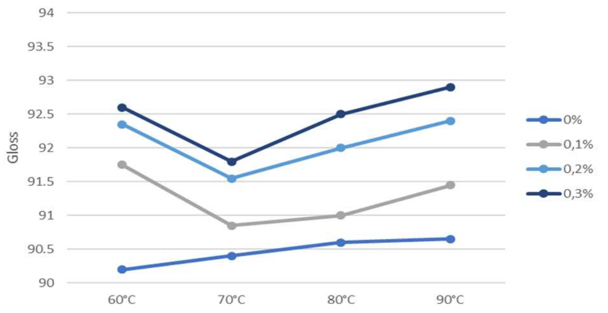
(c)



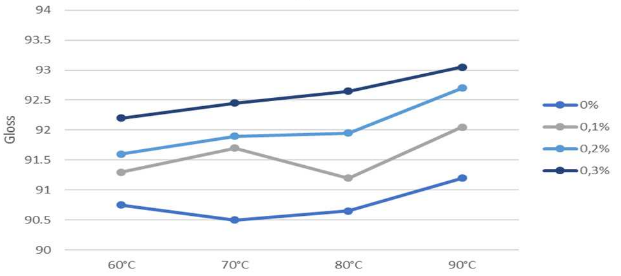
(d)

Fig. 1 Gloss Testing Results at Drying Temperatures of: (a) 60°C, (b) 70°C, (c) 80°C, (d) 90°C

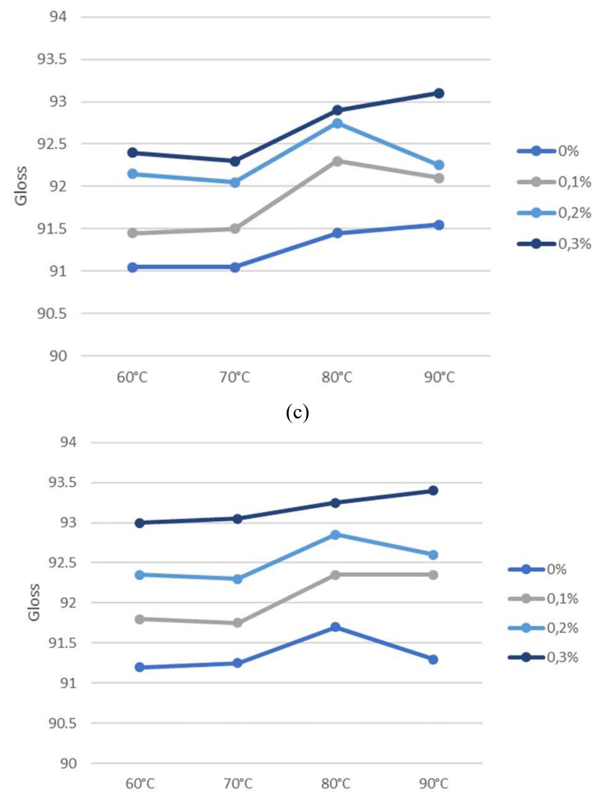
Fig. 2 (a)-(d) shows the gloss values for different concentrations of DBTDL at drying times of 15 minutes, 20 minutes, 25 minutes, and 30 minutes. From Fig. 1, it can be concluded that, generally, adding a DBTDL catalyst proved to increase the gloss value of the paint. Higher concentrations of the DBTDL catalyst resulted in higher gloss values. Fig. 2 also shows that, generally, gloss values increased with the increase in drying temperatures.



(a)



(b)



(c)

(d)

Fig. 2 Gloss Testing Results at Drying Time of: (a) 15 minutes, (b) 20 minutes, (c) 25 minutes, (d) 30 minutes

D. Sample Characterization

The following are the results of FTIR spectroscopy analysis conducted to observe the functional groups in the compounds under study qualitatively. This analysis helps identify the chemical structure and possible functional groups present in the samples. The FTIR identification was conducted by comparing the condition with 0% DBTDL as the standard, then compared with the addition of 0.1%, 0.2%, and 0.3% DBTDL. This was to identify functional group changes occurring after adding DBTDL in the polyurethane paint. Table 4 presents the FTIR analysis results at concentrations of 0%, 0.1%, 0.2%, and 0.3% [28].

Table 4 shows that adding 0.1% and 0.2% DBTDL does not result in significant changes in functional groups. However, in the C=O spectrum, there are differences in spectrum values, which may be due to the addition of DBTDL, as its molecular structure contains a C=O functional group. At a concentration of 0.3% DBTDL, there is a noticeable difference in spectrum values compared to the other samples, possibly due to reactions occurring with other compounds present in the polyurethane coating in the sample. A higher concentration can induce chemical interactions that result in the decomposition or transformation of these functional groups. Conversely, at concentrations of 0.1% and 0.2%, the amount of DBTDL may be insufficient to cause significant alterations in the FTIR spectrum, leading to results comparable to samples without DBTDL addition.

TABLE IV
FTIR SPECTRUM

	DBTDL 0%	DBTDL 0.1%	DBTDL 0.2%	DBTDL 0.3%	Functional groups
FTIR Spectrum (cm^{-1})	700.16	700.16	700.16	700.16	C-Cl or C-Br (alkyl halides)
	759.95	759.95	759.95	758.02	C-H (aromatic)
	1029.99	1029.99	1029.99	-	C-O (ethers or alcohols)
	1072.42	1072.42	1072.42	-	C-N (amines)
	1161.15	1161.15	1161.15	1165	C-C (cycloalkanes)
	1454.33	1454.33	1454.33	1456.26	C-H (alkanes)
	1724.36	1728.22	1726.29	1732.08	C=O (ketones, aldehydes, esters, or carboxylic acids)
	2958.8	2958.8	2958.8	2958.8	C-H (alkanes)

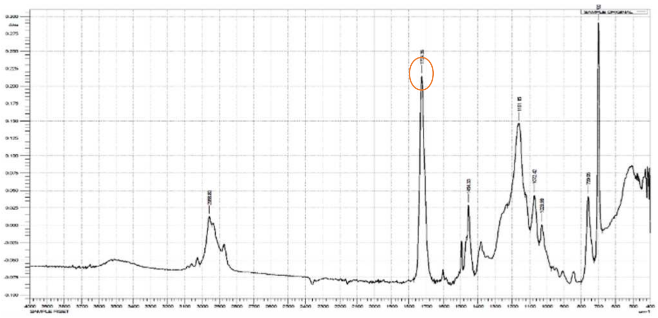


Fig. 3 FTIR at 0% DBTDL Concentration

The FTIR spectrum provided reveals the presence of various functional groups in the analyzed compounds. The peak at 700.16 cm^{-1} indicates the presence of C-Cl or C-Br bonds, characteristic of alkyl halides. The peak at 759.95 cm^{-1} suggests out-of-plane C-H stretching, typically found in aromatic molecules. The peak at 1029.99 cm^{-1} shows the presence of C-O bonds, common in ethers and alcohols, while the peak at 1072.42 cm^{-1} signifies C-N bonds, frequently found in amine groups. The peak at 1161.15 cm^{-1} is usually associated with C-C stretching, such as in cycloalkanes or esters. The peak at 1454.33 cm^{-1} indicates asymmetric C-H deformation in methyl or methylene groups found in alkanes. The strong peak at 1724.36 cm^{-1} is indicative of carbonyl groups (C=O), which can originate from ketones, aldehydes, esters, or carboxylic acids. Finally, the peak at 2958.8 cm^{-1} shows symmetric C-H stretching in methyl or methylene groups in alkanes.

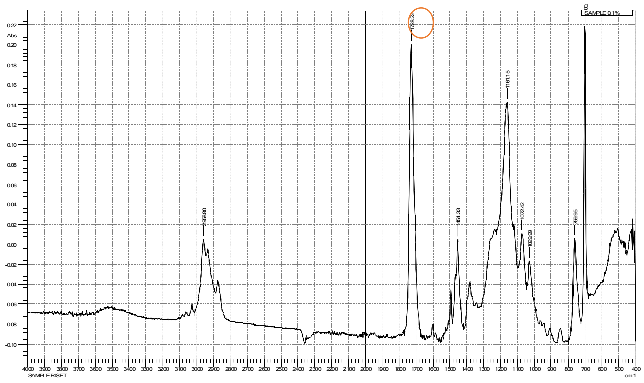


Fig. 4 FTIR at 0.1% DBTDL Concentration

The peak at 700.16 cm^{-1} indicates the presence of C-Cl or C-Br bonds, characteristic of alkyl halide groups. The peak at 759.95 cm-1 shows out-of-plane C-H stretching, commonly found in aromatic molecules. The peak at 1029.99 cm^{-1} indicates the presence of C-O bonds, often found in ethers and alcohols. The peak at 1072.42 cm^{-1} shows the presence of C-N bonds, typically found in amine groups. The peak at 1161.15 cm^{-1} is generally associated with C-C bonds, which can come from cycloalkanes or double bond stretching. The peak at 1454.33 cm^{-1} often indicates asymmetric C-H deformation in methyl or methylene groups, characteristic of alkanes. The peak at 1728.22 cm^{-1} strongly indicates carbonyl (C=O) groups, which can come from ketones, aldehydes, esters, or carboxylic acids. Finally, the peak at 2958.8 cm^{-1} shows symmetric C-H stretching in methyl or methylene groups in alkanes.

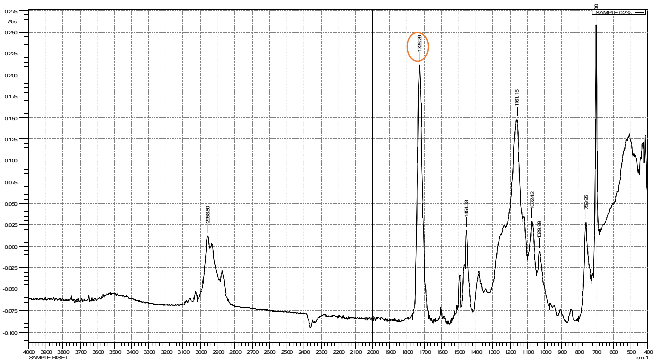


Fig. 5 FTIR at 0.2% DBTDL Concentration

The peak at 700.16 cm^{-1} indicates the presence of C-Cl or C-Br bonds, characteristic of alkyl halide groups. The peak at 759.95 cm^{-1} suggests the presence of out-of-plane C-H stretching, commonly found in aromatic molecules. The peak at 1029.99 cm^{-1} indicates the presence of C-O bonds, often found in ethers and alcohols. The peak at 1072.42 cm^{-1} signifies the presence of C-N bonds commonly found in amine groups. The peak at 1161.15 cm^{-1} is usually associated with C-C bonds, such as those in cycloalkanes or double bonds. The peak at 1454.33 cm^{-1} often indicates asymmetric C-H deformation in methyl or methylene groups, which is common in alkanes. The peak at 1726.29 cm^{-1} strongly indicates carbonyl (C=O) groups, which can come from ketones, aldehydes, esters, or carboxylic acids. Finally, the peak at 2958.8 cm^{-1} shows symmetric C-H stretching in methyl or methylene groups in alkanes.

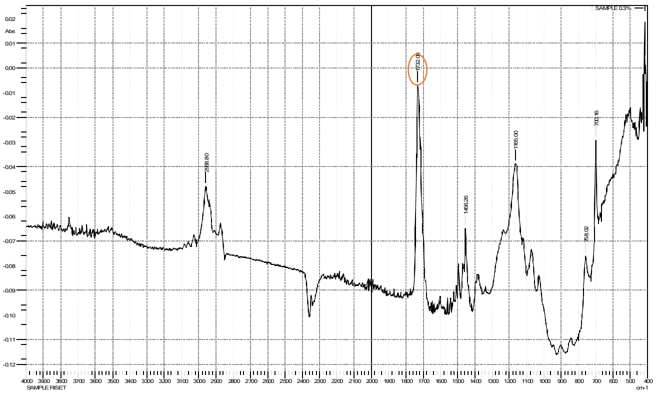


Fig. 6 FTIR at 0.3% DBTDL Concentration

The peak at 700.16 cm^{-1} indicates the presence of C-Cl or C-Br bonds, characteristic of alkyl halide groups. The peak at 758.02 cm^{-1} signifies out-of-plane C-H stretching commonly found in aromatic molecules. The peak at 1165 cm^{-1} is usually associated with C-O bonds, often found in esters or alcohols or C-C stretching in cycloalkanes. The peak at 1456.26 cm^{-1} frequently indicates asymmetric C-H deformation in methyl or methylene groups, which is common in alkanes. The peak at 1732.08 cm-1 strongly indicates carbonyl (C=O) groups, which can originate from ketones, aldehydes, esters, or carboxylic acids. Lastly, the peak at 2958.8 cm^{-1} shows symmetric C-H stretching in methyl or methylene groups in alkanes.

The shape of the FTIR graph shows consistent results at concentrations of 0%, 0.1%, and 0.2%. However, the results differ at a DBTDL addition concentration of 0.3%. At this concentration, the functional groups C-O (ether, alcohol) and C-N (amine) are absent in the sample. This likely occurs due to the polymerization reaction process during the sample mixing.

IV. CONCLUSION

DBTDL exhibits highly reactive properties and effectively accelerates the polymerization reaction, resulting in paint products that dry faster and are more durable. The research demonstrates that increasing the concentration of DBTDL in polyurethane paint yields good hardness and gloss values. However, in adhesion testing, a high concentration addition, excessively high temperatures, and prolonged drying times resulted in adhesion test results <100%. This was observed with a DBTDL addition concentration of 0.3%, temperatures of 80-90°C, and drying times of 30 minutes. The hardness, adhesion, and gloss tests achieved the optimal conditions with a concentration of 0.2% and a drying time of 30 minutes at temperatures of 80-90°C. In general, FTIR analysis, spectral changes were observed in the C=O functional group (ketones, aldehydes, esters, or carboxylic acids). Several functional groups were found missing at a 0.3% DBTDL addition concentration, possibly due to higher concentrations potentially triggering chemical interactions leading to degradation or alteration of these functional groups.

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