Yurtalan, N., Geyikçi, F., Uğuz, G. (2024) 7(2), 177-184.

RESEARCH ARTICLE



EFFECT OF BORIC ACID ADDITION ON ANTIBACTERIAL DYE PRODUCTION

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Abstract: In this study, the use of interior dyes with antibacterial properties was examined. Boric acid (H₃BO₃), which is a cheap and easily obtainable boron compound from our country's resources, has an antibacterial effect. Within the scope of the study, firstly, boric acid mixtures were prepared in appropriate concentrations and antibacterial dye production studies were carried out by adding them to indoor dyes. The antibacterial properties of the obtained dye were investigated through the inhibition effect on *E. coli* bacteria and its antibacterial activity was tested, and then the brightness, density, film thickness, scorching, pencil hardness and drying time tests of the dyes and the suitability of their characteristics to TS 5808/2012 and the applicability of these dyes were evaluated. The results showed that the antibacterial effect of boric acid did not have a negative effect on the dye quality, but maintained the standard values of the dye.

Keywords: Waste marble powder, Boric acid, Antibacterial material.

Submitted: June 21, 2024. Accepted: August 26, 2024.

Cite this: Yurtalan, N., Geyikçi, F., & Uğuz, G. (2024). Effect of boric acid addition on antibacterial dye production. Journal of the Turkish Chemical Society, Section B: Chemical Engineering, 7(2), 177–184. https://doi.org/10.58692/jotcsb.1503140

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1. INTRODUCTION

Boron and its compounds are frequently used in many industries. Due to the technology and its abundance, its applications are spreading over a wide area recently. Dyes are building materials that protect the surface of the material, provide an aesthetic appearance, and at the same time prevent factors such as rusting, contamination, corrosion, etc. It is a colored liquid material formed by the combination of metallic, organic, and plastic-based pigment, thinner and binding agents (Özkan, 2013).

Boric acid (H_3BO_3) is one of the most widely used boron compounds in industry as a source of B_2O_3 and is used in the preparation of many boroncontaining chemicals such as boron carbide and boron esters.

Additionally, it is used in antiseptics, boron alloys, fire retardants, nylon production, photography, textile industry, glass and glass fiber production, enamel, and glaze. In recent years, it has also found use as a super slider. Boric acid has a molecular weight 61.83 g/mol, B_2O_3 content of 56.3%, melting point of 171 °C, specific gravity of 1.44 g/cm³ is a crystalline substance with a temperature of formation of -1089 kJ/mol and a temperature of dissolution of +22.2 kJ/mol. Although its solubility in water is low at room temperature, its solubility increases significantly as the temperature rises. For this reason, it is generally considered sufficient to cool the saturated solution from 80 °C to 40 °C to crystallize boric acid in industry (Sarı, 2008; Kuru & Yarat, 2017).

The antibacterial property is sterilization and minimization of microbial action. Disinfectant materials are generally used to prevent this effect.

Disinfectants are chemicals used for places where pathogenic microorganisms exist or are likely to exist, and for devices or materials that may be a source of contamination. The pollution status of the environment in which the disinfectant will be used depending on many factors such as the type of microorganism, the surface to be disinfected, and the characteristics of the equipment, the characteristics of the disinfectant, and the cost (Abbasoğlu, 2007). Microorganisms are creatures that can be found everywhere and reproduce rapidly if suitable conditions are provided. An adequate supply of moisture, temperature, and nutrients is necessary for bacteria to reproduce. The optimal growth temperature of bacteria is between 30-37 °C, while for fungi this temperature is 25-30 °C. Regional temperature changes in the body are among the factors that trigger the proliferation of bacteria (Akaydin & Kalkanci, 2014).

Ensuring indoor air quality and removing surface contamination by using antibacterial materials are frequently applied. However, it does not provide assurance about decontamination due to reasons such as the use of water, detergents, and disinfectants creating difficulties. In reaching every area and the inability of the applied substances to provide a permanent effect. For this reason, coating large surfaces with antimicrobial materials are seen as a more permanent option to prevent crosscontamination caused by microorganisms such as objects, devices or walls that are at risk (Bal & Şanlı, 2020).

Boric acid is a hydrophobic element that is considered to be non-toxic if it does not reach systemically significant amounts in studies, especially useful for various systems. Especially, boric acid shows antibacterial, anticandidal, and antifungal activity. It has been shown in various studies to be an active antibacterial agent against bacteria such as *Staphylococcus aureus, Klebsiella pneumoniae, Candida albicans, Aspergillus Niger, Escherichia coli,* and *Pseudomonas aeruginosa* (Kapukaya & Külahçı, 2020; Zer, Karabacak, & Manay, 2022).

In different parts of the world, the research and development of new antibacterial agents effective against bacteria continues rapidly. Some researchers have focused on assessing the antibacterial effect of boric acid in this process (Haesebrouck et al. 2009; Concia et al. 2016).

It has been reported that the solution, which is formed by mixing 2% boric acid and 2% acetic acid proportions, in eaual inactivates S. *pseudintermedius* in the amount of 5×10^7 CFU/mL in 30 minutes when diluted at 1/2 and 1/4 ratios. It is stated that this effectiveness is increased more by the use of boric acid together with acetic acid. In a study conducted on humans in our country, it was reported that boric acid solution applied to the oral strong antibacterial effects on tract had Enterococcus faecalis (Zan et al. 2013)

Dyes can be divided according to their place of use and the type of binder in their content. Decorative dyes, construction dyes, industrial and corrosion dyes, furniture dyes, auto dyes, peelable dyes and food dyes are included in this classification (Çalışkan, 2019).

Especially in the Covid-19 pandemic we are experiencing, it has become clear how important hygiene is in public environment. Microorganisms can survive on surfaces for long periods of time, and contact with these surfaces can cause cross-infections. This is especially important in places such as food processing plants, offices, and hospitals (Bal & Şanlı, 2020).

In the selection of wall materials in hospital interiors, surfaces that do not emit dust, chemicals and vapors should be preferred. If the materials are fragmented, they should be used in large pieces and should not have a porous structure. In addition, it is recommended that the materials are easy to clean and have antibacterial properties (Kırbaş, 2012).

The use of antibacterial materials has been important in environments where the public can be found collectively such as indoor spaces, schools and hospitals, and where the microbial load is high. Antibacterial dyes and coatings have gained importance in order to prevent the growth and spread of these bacteria on surfaces such as walls, door handles, and tables. The production of antibacterial dye with boric acid, which is not harmful to human health, shows its usability in every possible environment. The emergence of products by using boric acid, which is abundantly available in our country, and our own resources can be considered as a national gain.

2. MATERIALS AND METHODS

2.1. Materials

Boric acid (H₃BO₃) was supplied by Eti Mining Enterprises. The contents of boric acid were represented in Table 1. Interior dye was supplied by Betek Dye and Chemical Industry Co. Inc. The features of interior dye were determined as 20 000410-TSE-115/01 standard and also were shown in Table 2. Antibacterial test kits supplied from (Hytech Slide) DiaTek Diagnostic Products Tech. Comp. Hytech Slide is a product that can perform analysis by taking samples from personnel, equipment, solid and liquid surfaces from all surfaces for microbiological analysis.

The storage temperature is 10 - 25 °C and the shelf life is 5-6 months. Each surface is divided into 10 areas ,each area being 1 cm². Thus, the results can be given as the number of colonies in a total surface area of 10 cm^2 .

Table 1. The contents of boric acid.

B ₂ O ₃	SO4 ²⁻	Cl⁻	Fe ²⁺
%59.25 min	500 ppm max	10 ppm max	7 ppm max

Table 2. Features of interior dye (20 000410-TSE-115/01).

Parameters	TS 5808
Brightness	Checkmate
Particle Size	Thin
Coating Power	Class 2 (7 m ² /L)
Wet Scrubbing Resistance	Class 3

2.2. Methods

2.2.1. Preparation of dye

Firstly, boric acid solutions were prepared in different concentrations (3%, 4%, and 5%). 85 mL of water-based dye was taken and diluted with 15 mL of pure water in three different experimental setups. 85 mL of the diluted dyes were taken and 15 mL of 3%, 4%, and 5% boric acid solutions were added to them. 100 mL boric acid added dyes were obtained by volume (% v/v).

2.3 Characteristic Dye Analyses

2.3.1 Antibacterial test

Water-based dye mixtures were prepared as 3%, 4%, and 5% boric acid solutions with distilled water and the antibacterial properties of mixtures were tested. 10 mL of homogenized dye and boric acid mixtures were taken and spread over a certain area and left to dry for 48 hours at room temperature. Similarly, only 10 mL of the reference sample, which is the dye, was taken and left to dry. The drying antibacterial dye is stained with 10⁸ Kob/g *E. coli* bacteria. The stained antibacterial dye samples were placed in the incubator to continue the bacterial activity.

The antibacterial properties of the surfaces were determined by taking tests with antibacterial test kits containing *E. coli* medium.

2.3.2 Brightness analysis

Luminance is a property related to the reflection of light falling on a surface, and the degree of luminance refers to the clarity of the image created on the surface by the rays from a source. Gloss measurement is carried out using photoelectric devices. The gloss depends on the number of binders and fillers of the dye, the particle size of the fillers (Uğuz, 2019).

2.3.3 Density analysis

. In addition to being an important parameter for calculating dye consumption, dye density is also important during the packaging of the product. Density measurement is generally carried out with TS EN ISO 2811-1 standard methods.

2.3.4 Film thickness analysis

1 gram of dye samples was applied on the aluminum surface (150 μm) and the thickness of the dye film samples was measured with the Loyka-5202-25 brand film thickness measuring device.

2.3.5 Adhesion analysis

The scanning method (squaring) is used. Areas of 1 $\rm cm^2$ are marked and divided into 100 equal parts by drawing 10 horizontal and 10 vertical lines that intersect each other perpendicularly with an interval

of 1 mm. It is adhered on an area of 1 cm^2 using adhesive tape. The tape is drawn perpendicular to the surface and the scorching dye areas are calculated as a percentage.

2.3.6 Pencil hardness analysis

It is used to determine the hardness of dye surfaces. In pencil hardness analysis, pencils from soft to hard are used: 5B, 4B, 2B, B, HB, 3H, 5H. With sandpaper, the tip of the pens is shaped into a square section. The degree of hardness, which is one level softer than the softest pencil that leaves a permanent mark on the dye surface by scratching the dye surfaces with pencils, is determined as the pen hardness of the film (Uğuz, 2019).

2.3.7 Drying time analysis

Samples are applied on the aluminum surface. 5, 10, 15, respectively. After minutes, the drying time is checked every 15 minutes for up to 4 hours, every 30 minutes after 4 hours, up to 8 hours and up to 24, 48 hours. First of all, it was tested whether the film sticks to our hands by touching our fingers, and after this stage, 1. For the degree of drying, a glass bead with a diameter of 0.2 mm was left on the dye surface from 5 cm above and it was checked whether it adhered to the surface. If it does not stick, the dye is 1. It has reached the degree of drying. Rounds 2 and 3 for drying degrees, the dye surfaces are covered with Kraft paper and masses of 0.05 N and 0.5 N, respectively, are applied for 1 minute and if the dye does not stick to the paper surface, the dye is 2 and 3. It has reached drying degrees. Rounds 4 and 5 at drying degrees, after the Kraft paper is placed on the dye surface, force is applied with masses of 5 N for 1 minute, if there is no adhesion but leaves a trace, the dye is 4. It reaches the degree of drying. If there are no traces and adhesions, the dye is 5. It reaches the degree of drying. Rounds 6 and 7 by applying the same processes within the drying degrees, a force weighing only 50 N is applied (Uğuz, 2019).

3. RESULTS AND DISCUSSION

3.1 Antibacterial Test

The obtained results showed that the effects of antibacterial dyes prepared at 3% and 4% concentrations on *E. coli* bacteria was less effective than the antibacterial dye prepared with 5% concentration. Since the percentage of boric acid will have a toxic effect after a certain concentration, optimum results have been obtained by keeping it at a concentration of 5%. In the boric acid dye mixture prepared with a concentration of 5%, the possibility of bacteria to survive on the surface decreased over time, and the antibacterial effect

decreased the number of *E. coli* over time. The surface of the test kits was divided into 10 areas of 1 cm^2 , and the number of bacteria on these areas was counted at the end of t=0, t=1, and t=2 hours, respectively. As a result of the count, it was seen that the number of live bacteria was active in 3 units of the 10-unit area divided. Antibacterial tests were carried out with the help of kits that tested the expected hygiene environment on the surfaces.

It is known that bacteria and disease-causing microorganisms are found in public areas. The fact of hygiene comes to the fore, especially during the Covid-19 period. The development of projects to prevent the spread of microorganisms are

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important, which is necessary for public health. In this reason, antibacterial dye using has been prioritized among the issues that need to be investigated. The low-cost antibacterial dye manufactured within the scope of the study can be easily applied to all areas (home, hospital, public transportation, etc.). It is envisaged that hygiene will be ensured indoors (especially in toilets). As can be seen in Figure 1, the antibacterial properties increase over time and the number of bacteria decreases.

In Figure 1, in the reference sample, bacteria are present in every 1 \mbox{cm}^2 area and continue their activities.

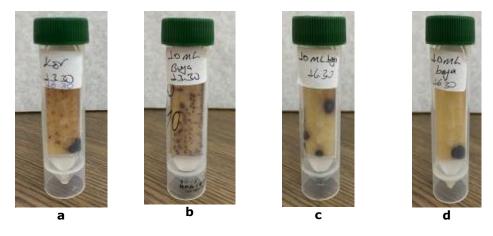


Figure 1. Antibacterial test results of samples (a) reference (b) 0 hour (c)1 hour (d)2 hours.

3.2 Analysis of Brightness

The results of the gloss analysis are given in Table 3. It is seen that the gloss values decrease with dilution and the contribution of boric acid.

Table 3. Change of gloss values in dyes.			
Samples	20°	60°	85°
Reference Dye	0.5 gloss	2.0 gloss	4.9 gloss
Diluted Dye	0.5 gloss	2.0 gloss	3.6 gloss
Boric Acid-Added Antibacterial Dye	0.4 gloss	1.9 gloss	3.1 gloss

3.3 Density Analysis

The results of the density analysis are given in Table 4. It is seen that the density values decrease with dilution and the contribution of boric acid.

Table 4: Change of density values in dyes.

Samples	Density (g/cm³)		
Reference Dye	1.6268 g/cm ³		
Diluted Dye	1.5568 g/cm ³		
Boric Acid Added Antibacterial Dye	1.4768 g/cm ³		

3.4 Film Thickness Analysis

The film thickness of the paints was measured with Loyka 5202-25 model micrometer. The results are given in Table 5.

Samples	Thickness (mm)	
Reference Dye	1.27 mm	
Diluted Dye	1.48 mm	
Boric Acid Added Antibacterial Dye	1.55 mm	

3.5 Adhesion Analysis

The results of the adhesion analysis for the dyes on the floor, which are given in Figure 2, are given in Table 6. Our dye samples (0) showed excellent adhesion by roasting.



Figure 2. Adhesion analysis experiment set.

Table 6. Change of roast values in dyes.

Roasting Degree	Blank Frames/ Total Frames		
Reference Dye	0/100		
Boric Acid-Added Antibacterial Dye	0/100		
Diluted Dye	0/100		

3.6 Pencil Hardness Analysis

The pencil hardness test set is given in Figure 3 and the analysis results are given in Table 7. There was no difference in film thicknesses.



Figure 3. Pencil hardness analysis experiment set.

Table 7. Change of pencil hardness values in dyes.

Samples	Degree of Hardness (g)	
Reference Dye	5B	
Diluted Dye	5B	
Boric Acid Added Antibacterial Dye	5B	

3.7 Drying Time Analysis

Drying time analysis results are given in Table 8. Drying degrees can be listed as Antibacterial dye < Dilute dye < Reference dye, respectively. The admixture of boric acid had little effect on drying.

Samples	2 h	4 h	8 h	24 h	48 h
Reference Dye	Sticks to the hand	Sticks to the hand	Grade 1	7th degree	7th degree
Dilute Dye	Sticks to the hand	Sticks to the hand	Sticks to the hand	Grade 5	7th degree
Boric Acid-Added Antibacterial Dye	Sticks to the hand	Sticks to the hand	Sticks to the hand	Grade 5	7th degree

Table 8. Change of drying time values in dyes.

4. CONCLUSIONS

Boron-based antibacterial dye was manufactured and the antibacterial activities of the dye samples were investigated. Traditional dye tests such as the degree of drying of the dye films, film thickness, gloss, and degree of spalling (adhesion) were carried out. The antibacterial dye properties produced are no different from the properties of the standard dve. Obtained results from characterization tests showed that the antibacterial dye did not lose its properties. There was no difference between standard dye and antibacterial dye in the drying degree, film thickness, gloss, roasting degree, density analysis. These dyes containing boric acid, which are generally used to dye the walls of hospitals, schools or crowded areas due to conditions that require hygiene, have gained antibacterial effectiveness from domestic and national resources and are promising for the dye market. It is predicted that the studies carried out are original within the scope of this information and will contribute to the antibacterial dye market and the literature.

5. ACKNOWLEDGEMENTS

This study were supported by Ondokuz Mayıs University BAP Unit with PYO.MUH.1908.22.059 Project Number. The authors are thankful for Dr. Nilgün ÖZDEMİR in Ondokuz Mayıs University Food Engineering Department for Antibacterial Analysis.

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