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作品名稱 **Synthesis of Macro Porous Activated Carbon from Waste Polyethylene Terephthalate (PET) Bottles and Investigation of Usability in Dye Removal from Water Sources**

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關鍵詞 **Waste Polyethylene Terephthalate、Activated Carbon、Methylene Blue**

作者照片

Project Name:

Synthesis of Macro Porous Activated Carbon from Waste *Polyethylene Terephthalate* (PET) Bottles and Investigation of Usability in Dye Removal from Water Sources

OBJECTIVE:

Colorants are used in many industries, especially in the textile industry. These substances both cause visual pollution and create an anaerobic environment for aquatic creatures. In this study, it is aimed to examine the usability of activated carbon synthesized from waste *polyethylene terephthalate* (PET) bottles, which is an important environmental problem, in removing the pollution caused by the colorants caused by industrial activities in water resources.

1. INTRODUCTION

Technological development and increasing consumption, industrialization, urbanization, population growth due to development in technology; causes the occurrence of environmental pollution problems. Wastes have become the most important problem nowadays and solid wastes are a cause of concern having a large share among these. Solid wastes are divided into domestic wastes and industrial wastes (Solak and Pekk Küçükşen, 2018). Over the years, plastics have become an integral part of our society with their superior features such as adaptability in design, thermostability, transparency, low cost and easy use. These plastics, which are used as packing and packaging materials, create a solid waste problem immediately upon their use. Among the total plastic wastes, the biggest share belongs to the domestic sourced packaging wastes with 40%. One of the most used types of plastic materials mentioned is *polyethylene terephthalate*, in other words PET. Especially *polyethylene terephthalate* (PET) consumption has the fastest growth rate in the global plastics market due to the ever-increasing expansion of the PET bottle market. In our country, approximately 165 thousand tons of PET bottles are produced annually. PET wastes do not disappear for many years due to its high resistance to natural conditions (Tayyar and Üstün, 2010). Plastic wastes are divided into small pieces over time and can be mixed with water by turning into microns. Thus, it poses a danger to living creatures as a result of its inclusion to the food chain with both underwater creatures and dangerous organic substances adsorbed by plastics (Yurtsever, 2019). In addition to micro-scale pollution, plastics also create serious problems on the macro scale and create aesthetically unpleasant waste areas. For these reasons, studies on the recycling and evaluation of PET wastes have gained importance today (Waste Management Pyramid, http://www.pagcev.org/geri-donusum).

Many countries establish waste policies regarding the recycling and recovery of such wastes. One of these policies is the reintroduction of PET wastes through recycling. There are three basic principles of solid waste management domestically and globally:

- Less waste production,
- Recycling of wastes,
- Disposal of wastes without harming the environment (Turkish Ministry of Environment and Urbanization, 2017).

Recycling of plastic wastes and developing new technologies for recycling are important in terms of finding solutions to increasing environmental problems by reducing the volume of waste, but it will also be an important gain for the country's economy (Tayyar and Üstün 2010). Synthesizing activated carbon from carbon-rich wastes such as *polyethylene terephthalate* (PET) is recommended as one of the most environmentally friendly solutions for waste recycling (Esfandiari et al., 2012).

In this study, it was aimed to investigate the use of activated carbon synthesized from *polyethylene terephthalate* (PET) bottles in removing the pollution caused by the colorants generated by industrial activities in water resources. For this purpose, methylene blue adsorption efficiency of activated carbon synthesized from waste PET bottles was investigated.

1.1 *Polyethylene terephthalate* (PET)

PET is a linear polyester produced by condensation polymerization of ethylene glycol with terephthalic acid or dimethyl terephthalate. The connection between units is in the form of ester bonds, it is a polymer that is not affected by acids but is sensitive to alkalis (Ministry of Environment and Urbanization, 2017). The chemical *formula* of polyethylene terephthalate is shown in Figure 1. In table 1, physical and chemical properties of *polyethylene terephthalate* (PET) are shown.

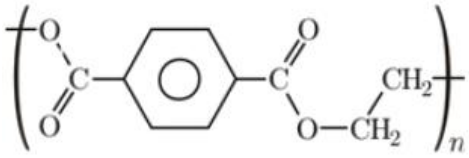


Figure 1. Chemical formula of *polyethylene terephthalate* (Anabal, 2007).

Table 1. Physical and chemical properties of PET (Anabal, 2007).

Physical/Chemical Property	**Value**
Molecular Weight, g/mol	192
Density (g/cm^3)	1.41
Expansion Coefficient, K^{-1}	$7x10^{-5}$
Glass Transition Temperature, °C	69-115
Melting Temperature, °C	265

The usage areas of PET material, which we may encounter in many places in the industrial field, are increasing day by day. The development of PET started in the 1950s. It was first produced in the form of a film, then took its current shape in the 1970s with air blow-molding processes. Due to its light weight, it is frequently used in the packaging industry as an alternative to glass. Today, it is known that it is used as a raw material instead of glass in the production of photographic films, textiles, audio-videotapes, electrical insulation and packaging. The most common usage areas are fiber and packaging industries. The production of polyester fiber, an important material of textile, accounts for 60% of PET usage. Another important area of use is the packaging industry. As packing, it is used in the packaging of many products from drinking water to cleaning materials, from foodstuffs to soft drinks. Nowadays, PET bottles have been used frequently due to the increase in population and consequently consumption. The ease of use they provide is an important factor in their preference. At the same time, many features such as being easy to process, not creating odor, being transparent, not having the risk of breaking, being light, being physically and chemically resistant, increase their likeliness of usage multiple times (Saçak, 2002; Anabal, 2007).

In addition to the advantages mentioned above, PET has some disadvantages such as low water holding capacity, low dyeability, high static loading and poor adhesive properties (Aşkın, 2007). PET is a thermoplastic material. Thermoplastic materials soften under heat and pressure and can be shaped and dissolved in suitable solvents (Şahmetlioğlu, 2000). Therefore, waste PET bottles can be recycled with a heating process above glass transition temperature. In addition, instead of recovering the same product with recycling, a different product or energy can be obtained by this process. Thanks to its high carbon content, PETs are very suitable waste materials for active carbon production (Bulak, 2011).

1.2 Activated Carbon

Activated carbon is a processed carbon substance that has the ability to adsorb many substances from gas and liquid phase due to its advanced porous structure and large internal specific surface area (Jankowska et al., 1991; Othmer, 1992). Activated carbon is produced from all materials containing carbon by chemical or physical activation. Since they have high carbon content, high surface area and pore structure, their adsorption efficiency is quite high. For this reason, activated carbons have been used as adsorbents for refinement long since (Arena & Clift, 2016). The properties of activated carbons can be listed as follows:

- ✓ It is an environmentally friendly adsorbent.
- ✓ It is the only commercial adsorbent that can be used in dehumidification and purification studies.
- ✓ It is very useful as it has high pore and inner surface area.
- ✓ It absorbs more organic matter than other materials used for similar purposes (Kaçan, 2011).

The use of activated carbon was used for the first time by Hippocrates in the form of charcoal to remove unpleasant odors. It is also known to be used in the treatment of diseases such as epilepsy and anthrax (Aygün, 2002). Its first industrial use was carried out by the Swedish chemist Karl Wilhelm Scheele in the 18th century by gas adsorption using charcoal. It has also been used frequently as a color remover. For example, Lovits, a Russian academic, bleached the homogeneous mixture of tartaric acid containing organic matter using charcoal (Stoeckli and Kraehenbuehl 1984). Whereas the first powder activated carbon was used in Europe in 1909; the first activated carbon was used in United States in 1913.

As a result of the I.World War, activated carbon was used in gas masks for a vital purpose. These activated carbons were produced from coconut raw material. After the war, it continued to be used in areas such as tap water treatment and sugar beet industry. In recent years, it has been used in the recovery of some metals (gold, silver, molybdenum, etc.) named as hydrometallurgy (Aygün, 2002).

The properties of activated carbons, which can be produced from all materials containing carbon, vary depending on the type and structure of the raw material used (Teng et al., 1996). Regarding the selection of the material to be used in the production of activated carbon, criteria such as the presence of the material in nature or as industrial waste and its low cost are taken into consideration. Today, for this purpose, many materials have been used for activated carbon production.

Some of these can be listed as follows:

- Polymers (Yue et al., 2002),
- Almond peels (Toles et al. (2000),
- Apple pulp (Suárez-Garcıa et al., 2002),
- Sugarcane bagasse (Mohan and Singh, 2002),
- Charcoal (Kütahyalı and Eral 2004), Peach kernels (Attia et al., 2008),
- Peanut shell (Girgis et al., 2002),
- Olive seed (Michailof et al., 2008; Kütahyalı & Eral, 2010),
- Coconut shell (Tay et al., 2001),
- Rice shell (Guo & Rockstraw, 2007),
- Polymer waste (Youssef et al., 1994),
- Waste generated as a result of agricultural activities (Sud et al., 2008),
- Date seeds (Haimour and Emeish, 2006),
- Waste car tires (Yakaboylu, 2010),
- Cotton stalk (Bolgaz, 2008),
- Palm bark (Özdemir, 2011).

1.2.1 Activated Carbon Synthesis

Activated carbon production takes place by subjecting used organic or inorganic materials to carbonization and activation processes. The carbonization process is the oxygen-free burning process of the substance in an oxygen-free environment without using chemicals. The purpose of this process is to shape the initial porous structure (Teng et al., 1996). The activated carbons obtained as a result of this process generally have a low surface area and this is not sufficient for the adsorption process. Activation process is required to increase the surface area. There are two main activation processes. These are physical and chemical activation processes. CO, CO_2 gases and water vapor are used in the physical activation process. If during activation process using gases, the temperature is 800-1000°C, the reaction rate increases (Kirk-Othmer, 1997). Chemical activation is performed using chemical agents such as zinc chloride ($ZnCl_2$), potassium sulfur (K_2S), potassium thiosionate (KSCN), sulfuric acid (H_2SO_4) and phosphoric acid (H_3PO_4) on non-carbonized material (Kütahyalı, 2002). After the activation process with a chemical agent called impregnation, the material is carbonized at 500-800°C in an inert environment inside a tube furnace under nitrogen gas flow (Jankowska, 1991). After this process, the activation agent is removed from the ambient by washing. Activated carbon may contain other elements than carbon in its structure. The ingredients it contains varies according to the raw material used, but generally contains oxygen, hydrogen and trace amounts of sulfur and nitrogen. It also contains 20% ash or residual minerals formed during combustion. Table 2 shows the proportions of the elements
% contained in activated carbons. (Ullmann's, 1986).

Table 2. Composition of activated carbons (Ullmann's, 1986).

Content	**Composition %**
Carbon	80-95
Hydrogen	0.6–3
Oxygen	1-8
Nitrogen	0–0.6
Sulphur	0–0.3

1.2.2 Usage Areas of Activated Carbon

Activated carbon is used as an important component in many areas due to its high surface area and porous structure. Some of the usage areas of activated carbon can be listed as follows (Tadda et al., 2016):

- Recovery processes,
- Purification of domestic and industrial waste water,
- Separation and purification of gases,
- Gas masks,
- Air conditioning systems,
- Paint removal from industrial waste water,
- Separation of unwanted substances from aqueous and organic solutions,
- Adsorption.

1.3 Adsorption

The attachment of atoms, ions or molecules to a solid surface is called adsorption. The solid material used during the adsorption process is called adsorbent, and the substance attached on the solid surface is called adsorbed. Adsorption occurs spontaneously at constant temperature and constant pressure (Sarıkaya, 1993). This process is in the form of substances dissolved in liquid phase adhering to a solid adsorbent surface. Adsorption is a frequently preferred method in recent years because it enables the separation of trace elements from large volume solutions at low cost (Berkem and Baykut, 1977).

2. MATERIALS AND METHODS

2.1. Chemicals, Devices Used in the Study and Fields of Use

Chemicals and devices used in this experimental study are given in Table 3.

Table 3. Chemicals, devices and fields of use

Used chemicals and devices	**Fields of Use**
Mettler Toledo pH meter	Measuring the solution pH
GFL 1086 temperature-controlled thermostat shaker	Performing methylene blue adsorption
Sartorius CP224S electronic precision balance	Weighing process
Binder oven	Drying process
Sinbo grinder	Shrinking PET bottle parts
Ceramic crucible (4x12,5x2)	Activated carbon synthesis
UV Visible spectrophotometer (1601Schimadzu)	Determining color solution concentration
Methylene blue (C16H18ClN3S) (Merck)	Determining the reception capacity

Tube furnace (Protherm)	Activated carbon synthesis
Zinc chloride ($ZnCl_2$) (Merck)	Activation agent in chemical activation process
Nitrogen gas (N_2) (Güneş Gaz Distribution)	Providing an inert environment in the PROTHERM tube furnace
Magnetic stirrer heater (MTOPS MS300HS)	Performing impregnation with ZnCl2

2.2. Activated Carbon Synthesis

Activated carbon synthesis from waste *polyethylene terephthalate* (PET) bottles was performed by chemical activation method, taking into account the studies of Kaur et al., 2019; László and Szucs, 2001. First, the waste PET bottles were cut into small pieces and passed through a grinder into smaller pieces (2 mm) (Figure 2). After the grinding process, pre-carbonization process was performed under N_2 gas using a PROTHERM tube furnace at 700°C for 30 minutes (Figure 3). Figure 4 shows the process setup.

Figure 2. Waste *polyethylene terephthalate* (PET) bottles after grinding.

Figure 3. After pre-carbonization process (700°C, 30 minutes, N_2).

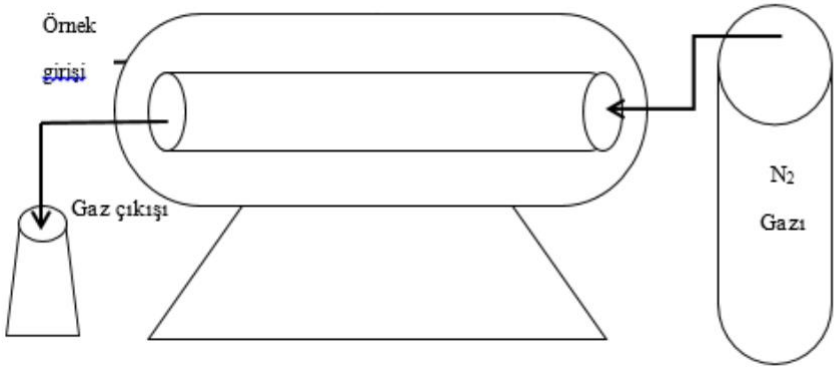


Figure 4. PROTHERM tube furnace process setup.

$ZnCl_2$ was used as a chemical reagent to activate the carbonized material. In order to observe the effect of the activation agent ratio, two samples of $ZnCl_2$ carbonized PET pieces were prepared at the ratio of 1/1 and 1/2, and they were mixed in a magnetic stirrer at 30 rpm (RPM) for 24 hours and subject to impregnation process. Figure 5 shows the impregnation process. After the activation process, the samples were filtered and left to dry overnight in the furnace at 105°C. The carbonization process was applied under N_2 gas at 800°C for 1 hour using PROTHERM tube furnace and at the end of the duration it was allowed to cool under N_2 gas. After the cooling process, the washing process was initialized and the activated carbons obtained were washed with 10% and 0.5 M HCl, hot distilled water and cold distilled water, respectively, until no chloride remained. The chloride test was carried out using $AgNO_3$ solution. Drying was carried out in an oven at 105°C. The flow chart of activated carbon synthesis from waste PET bottles is given in Figure 6. The synthesis reaction yield was determined as 8.18±0.5%.

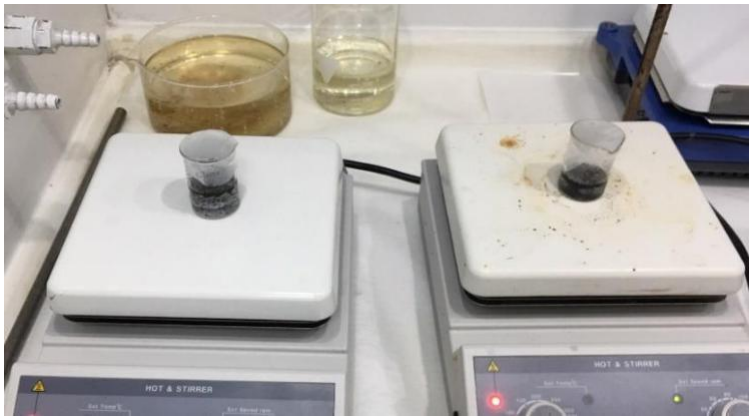


Figure 5. Impregnation process using $ZnCl_2$

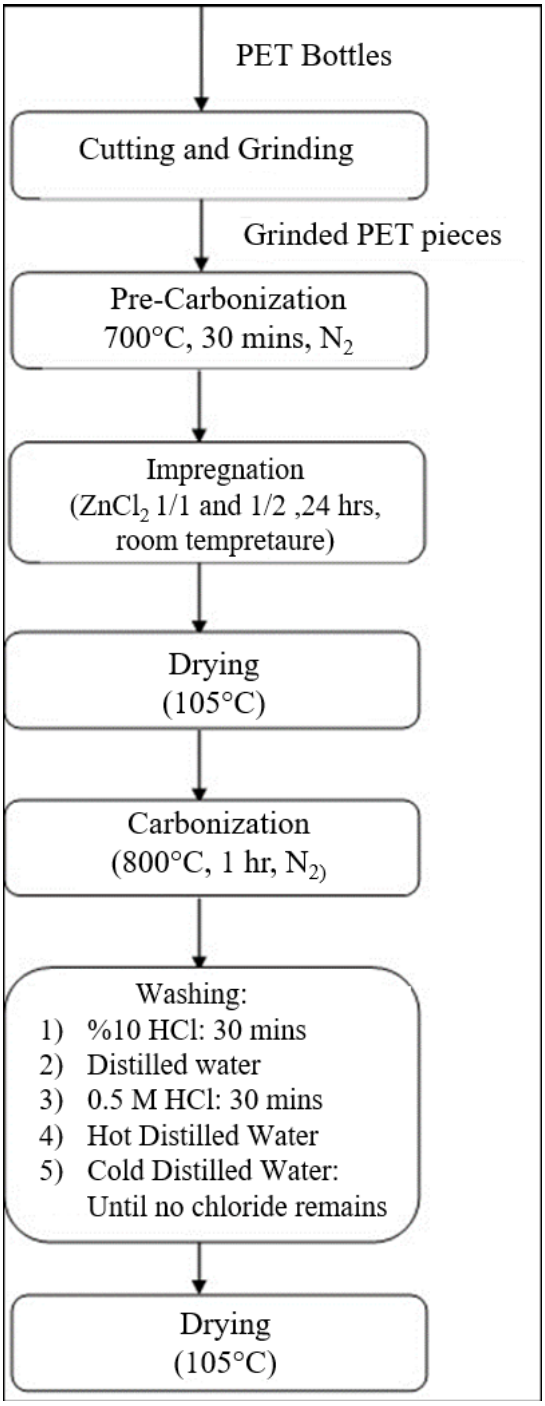


Figure 6. Flow chart of activated carbon synthesis from waste PET bottles.

3. RESULTS

3.1 Determination of Uptake Capacity of Synthesized Activated Carbon

0.01 g of activated carbon was added and shaken at 25°C for 24 hours to 50 ml of methylene blue solutions with a pH of 4.72 at 1000 mg/L using a glass erlenmeyer to determine the uptake capacities of activated carbon samples synthesized in the ratio of 1/1 and 1/2 ($ZnCl_2$ / carbonized PET pieces) from waste PET bottles. At the end of the shaking process, the samples were filtered and the concentration of methylene blue remaining in the solution was determined with a UV Visible Spectrophotometer (1601 Schimadzu) device at a wavelength of 664 nm (Figure 7). For this purpose, the calibration graph plotted against the absorbance values in different concentrations of methylene blue was used. According to the analysis results, the intake efficiencies were calculated as 22.4% for activated carbon synthesized at the rate of 1/1 and 1.2% for the activated carbon synthesized at the rate of 1/2, using equation (1), respectively. It is seen that activated carbon synthesized at the rate of 1/1 according to their intake yields has a higher efficiency, and subsequent studies were carried out with this material called PET-AC.

$$\%\boldsymbol{Uptake\ Efficiency} = \frac{\boldsymbol{C_0 - C_e}}{\boldsymbol{C_0}} \times \mathbf{100} \qquad (1)$$

Here; C0is: Initial solution concentration, Ce is: The amount of adsorbate remaining in solution in equilibrium (mg/L).

Figure 7. UV Visible Spectrophotometer (1601 Schimadzu) device.

3.2 Characterization of PET-AC Material

3.2.1 SEM and SEM-EDS Analysis

In order to examine the surface morphology of activated carbon synthesized from waste PET bottles, SEM (Scanning Electron Microscope) analysis was performed using the Thermo Scientific Apreo S branded device in EGE MATAL. SEM image of PET-AC material is given in Figure 8. PET-AC activated carbon synthesized from waste PET bottles appears to have a porous surface. Thus, it can be interpreted that the activation agent ($ZnCl_2$) used in the impregnation process reacts not only with the surface but also with the material gradually by passing through the inner parts of the material (Esfandiari et al., 2012; László and Szucs, 2001). Based on IUPAC (International Union of Pure and Applied Chemistry) standards, adsorbents are classified as <2 nm micro-pores, 2-50 nm mesopores and> 50 nm macropores according to their pore sizes (Bolgaz, 2008).

According to this classification, it is seen that PET-AC material synthesized in this study has macro porosity (Figure 8).

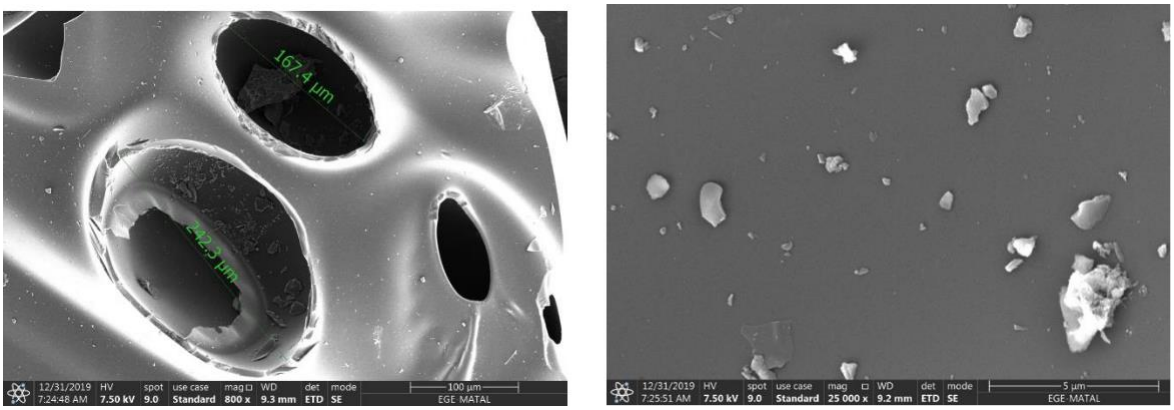


Figure 8. SEM image of PET-AC material.

SEM-EDS (Energy Distribution Spectrum) analysis was applied for PET-AC and elemental analysis was performed (EGE MATAL-Thermo Scientific K-Alpha device). The elemental content of the material is listed in Table 4. Figure 9 shows the SEM-EDS spectrum for PET-AC.

Table 4. PET-AC material element content

Element	**% by mass**
Carbon	92.04
Oxygen	2.74
Sulphur	5.22

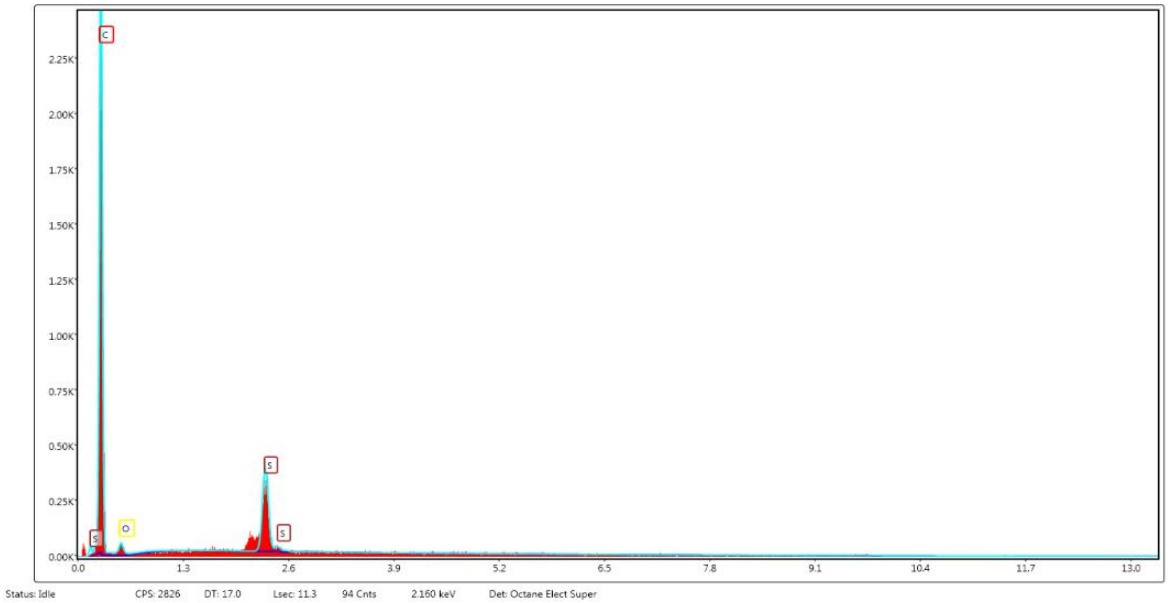


Figure 9. PET-AC SEM-EDS analysis spectrum.

3.2.2 Specific Surface Area Analysis

Specific surface area analysis was performed by applying the methylene blue adsorption data to the Langmuir adsorption equation for activated carbon (PET-AC) adsorbent synthesized from waste PET bottles, considering the work of Tewari and Thornton, 2010. Trials were carried out with synthesized PET-AC with methylene blue adsorption at concentrations varying between 1-150 mg/L. The Langmuir isotherm is calculated using equation (2):

$$\frac{\mathbf{1}}{\boldsymbol{q_{eq}}} = \frac{\mathbf{1}}{\boldsymbol{Q^0}} + \frac{\mathbf{1}}{\boldsymbol{C_{eq}}} \times \frac{\mathbf{1}}{\boldsymbol{bQ^0}} \qquad (2)$$

Here, Ceq is: The amount of adsorbate remaining in equilibrium solution without adsorption (mg/L), qeq is: The amount of adsorbate adsorbed by 1 gram of adsorbent (mg/g), Q^0 and b is respectively the reciprocal of the maximum adsorption value and the concentration at which half-saturation of the adsorbent is achieved.

The data calculated in obtaining the Langmuir isotherm are listed in Table 5. Figure 10 shows the linear Langmuir isotherm. The high correlation coefficient of the Langmuir isotherm (R^2=0.9893) confirms that the adsorption of methylene blue on PET-AC takes place in a single layer.

Table 5. PET-AC material element content

Ce (mg/L)	qe (mg/g)	1/Ce (L/mg)	1/qe (g/mg)
1.11E-04	1.00E+01	9.01E+03	1.00E-01
2.05E-04	2.00E+01	4.88E+03	5.00E-02
4.52E-04	4.00E+01	2.21E+03	2.50E-02
6.27E-04	6.00E+01	1.59E+03	1.67E-02
1.19E-03	1.00E+02	8.40E+02	1.00E-02
2.46E-03	1.05E+02	4.07E+02	9.55E-03
3.74E-03	1.05E+02	2.67E+02	9.54E-03
3.58E-02	1.23E + 02	2.79E+01	8.14E-03

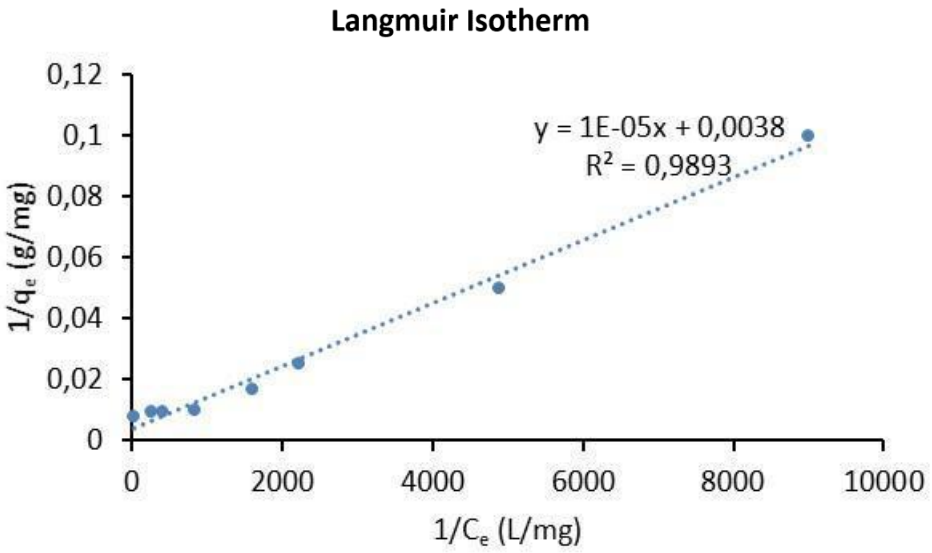


Figure 10. Langmuir isotherm in the adsorption of methylene blue on PET-AC.

Specific surface area is calculated using equation (3):

$$Specific\ Surface\ Area\left(m^2/g\right) = X_m.N.A \qquad (3)$$

Here; Xmis: intake capacity (mol/g) of additional Tlayer, N is: Avogadro number and A is: Methylene blue adsorbed area per molecule (130 $Å^2$) (Tewari and Thornton, 2010).

In the specific surface area analysis, the Xm value was obtained from the adsorption isotherm corresponding to the inflection point obtained from the drawing of the Ceq versus qeq graph. Figure 11 shows the graphic of the isotherm. In the light of this information, the specific surface area of activated carbon (PET-AC) synthesized from waste PET bottles was calculated as 206.46 m^2.

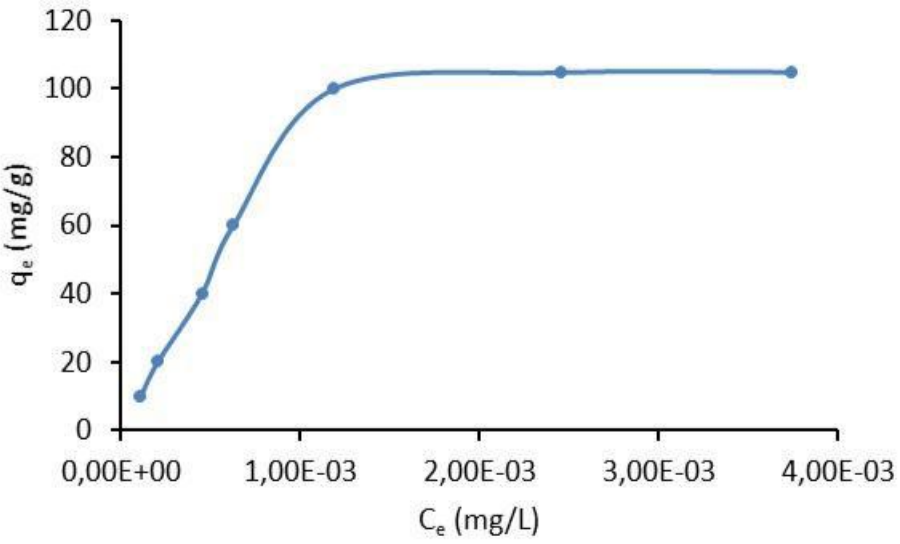
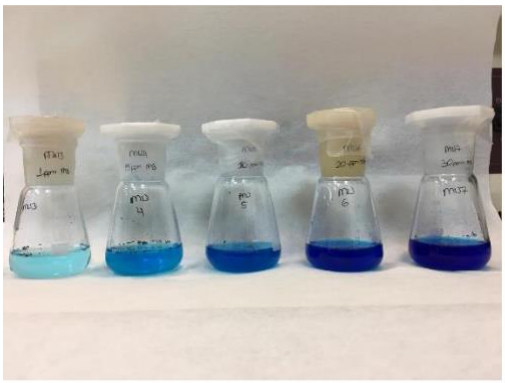


Figure 11. Adsorption isotherm of methylene blue on PET-AC.

3.3 Effect of Concentration on Methylene Blue Adsorption on PET-AC

In order to examine the effect of concentration on the adsorption of methylene blue on PET-AC, methylene blue solutions varying between 1-150 mg/L were added to 0.01 g adsorbent and agitated at 25°C for 24 hours. The methylene blue solutions before and after adsorption are shown in Figure 12 and the working setup in which the experiments are carried out are shown in Figure 13.

A



B

Figure 12. Methylene blue solutions before (A) and after (B) adsorption.

Figure 13. Water bath-thermostat shaker for adsorption trials.

After the shaking process, the samples were filtered, and the concentrations of methylene blue were adjusted to 664 nm with the UV-Visible Spectrophotometer device. The intake yield results are given in Table 6. The effect of concentration on the uptake efficiency in methylene blue adsorption of PET-AC is shown in Figure 14. When the intake yields were compared, it was concluded that the synthesized PET-AC adsorbent material was an effective material in terms of removing the impurities caused by dyestuffs in water sources with 94.07% uptake efficiency observed at a concentration of 50 mg/L methylene blue.

Table 6. Effect of concentration on methylene blue adsorption by PET-AC

Methylene Blue Concentration (mg/L)	Intake Efficiency (%)
1	83.26
5	72.27
10	74.36
20	71.73
30	73.86
50	94.07
100	93.86
150	83.40

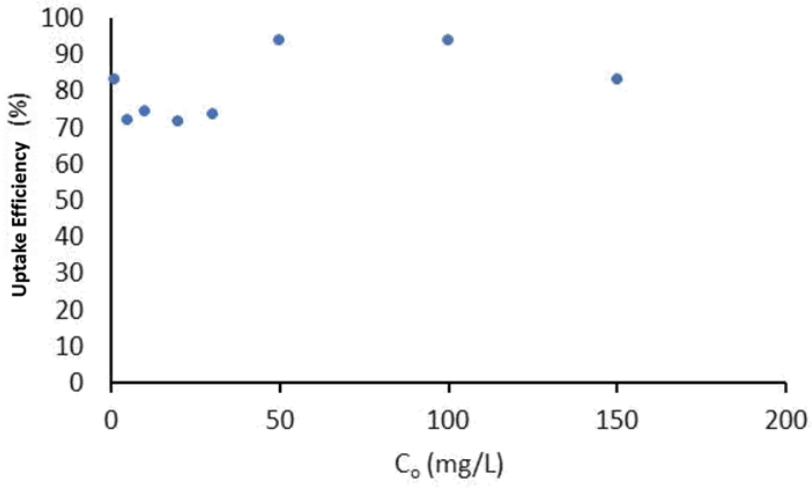


Figure 14. Effect of concentration on the adsorption of methylene blue on PET-AC (pH 4.72, Temperature 25 °C and shaking time 24 hours).

4. CONCLUSION AND DISCUSSION

In our study, *two separate activation agent* ($ZnCl_2$)/material ratio (1/1 and 1/2) activated carbon was synthesized from waste polyethylene terephthalate (PET) bottles by chemical activation method. The synthesis efficiency of this process was calculated as 8.18±0.5%. The uptake efficiencies of the synthesized activated carbons were determined by performing methylene blue adsorption; The intake yields were calculated as 22.4% for activated carbon synthesized at 1/1 rate and 1.2% for activated carbon synthesized at a rate of 1/2, respectively. SEM, SEM-EDS and specific surface area analyzes were performed for the characterization studies of activated carbon (PET-AC) synthesized at 1/1 ratio from waste PET bottles. As a result of the analysis, it was observed that PET-AC has a porous structure, and the specific surface area calculated by forming the Langmuir isotherm was 206.46 $m.^2/g$. Solution pH 4.72, ambient temperature 25 ° C and shaking time 24 hours conditions methylene blue concentration; The effect of methylene blue on the adsorption of PET-AC was investigated and PET-AC uptake efficiency was calculated as 94.07%. With its high intake efficiency, it was concluded that the activated carbon synthesized in order to be used in the removal of the pollution caused by the dyestuffs formed as a result of industrial wastes in water resources is an effective material. One of the advantages of the study is that waste *polyethylene terephthalate* (PET) bottles, which are an important environmental problem, are used as raw materials in activated carbon synthesis.

5. RECOMMENDATIONS

Considering the studies conducted, we believe that PET-AC, which is thought to be an effective adsorbent in dye removal, can be used in waste treatment facilities. In addition, it is considered important to examine the use of PET-AC adsorbent in the removal of other contaminants (inorganic and organic) and to determine its effectiveness.

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PET recycling is a very critical action worldwide. However, the use of recycled PET is limited. The student presented a possibility for use as activated carbon. The idea is interesting. However, the scientific depth is limited and more studies could tell us the true potential of this application.