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Performance of Gas-phase Toluene by Adsorption onto Activated Carbon Prepared from *Robinia pseudoacacia* L. as Lignocellulosic Material

Kaan İŞINKARALAR*¹

Abstract

The main target of this study was to eliminate gas-phase toluene with activated carbon from indoor air. The activated carbons were prepared from *Robinia pseudoacacia* L. biomass under different conditions. The change in surface functional groups of the activated carbon biomass raw material produced by pyrolysis in the absence of oxygen at 500–900 °C and activation by potassium hydroxide (KOH). The highest surface area of 1271.3 m²/g gives reason for its external porous surface. The surface porosity and the graphite properties of the prepared KN_xACs were detected by scanning electron microscope (SEM). The amount of adsorbed toluene (C₇H₈) was determined using a gas chromatograph-mass spectrometry with a thermal desorber system (TD–GC–MS) on the KN_xAC surface. The toluene adsorption capacity was reached 111 mg/g at 25 °C and 1000 ppm. As a result, the study revealed that the prepared KN₂₄AC from the *Robinia pseudoacacia* L. biomass has the best adsorption capacity of gas-phase toluene from indoor air.

Keywords: Activated carbon, lignocellulosic material, indoor air quality, toluene removal

1. INTRODUCTION

Volatile organic compounds (VOCs) have high vapor pressure easily releasable to indoor air [1-3]. Toluene (C₇H₈) is commonly found in indoor air as VOC, affecting human health [4-6]. It is prevalent in the synthesis of chemicals, petroleum refining, wood goods, and several industries' activities [7-9]. The gas-phase toluene can cause several problems, such as photochemical smog in the winter season, adverse effects on plants, and human diseases (respiratory, nervous, etc.) [10-13]. The availability of toluene in industrial air is less than 265.76 ppm, although many industrial processes cause to limit the excess of this value [14]. World Health Organization (WHO) reported

that the concentration of toluene should not exceed 53.15-106.3 ppm due to adverse effects on human health. According to the Bureau of Indian Standards (BIS) should be 200 ppm for the threshold limit of intake. On average, an employee is exposed to approximately 200 ppm for 8 hours a day in offices, industries, etc. Therefore, removing gas-phase toluene from indoor air is crucial to block its adverse impact on indoor air quality [15-18].

There are various methods in use to remove gaseous pollutants, such as physical (absorption and adsorption), chemical (scrubbing and oxidation), and biological (bio-filter and bio-scrubbing) ways [19-21]. Among them,

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adsorption has been widely used to remove VOCs from the air [22-24]. The advantages of adsorption are included low operational cost, easy accessibility of used materials, sometimes reusability, and high removal efficiency [25]. In particular, the performance of adsorption can easily increase the efficiency with adsorbate structure and properties [26]. The most widely used adsorbate is activated carbon with numerous pore sizes (including micro and macropores) [27]. Carbon-based adsorbents' efficiency and success in removing VOCs have made them more familiar and preferable materials [28]. The most distinctive feature of a high affinity is the result of the interaction of the non-polar adsorbent and the adsorbate [29]. For the adsorption process to work with high efficiency, it aims to develop the raw material and production method of the adsorbent. Thanks to this, it has an important place in the adsorption of gases due to obtaining a high surface area and improving the micropore volume.

Lignocellulosic materials such as *Tamarix hispida*, *Diploaxis harra*, *Glebionis coronaria* L., apricot kernels, date palm seed, and coconut shells are plentiful, and many investigations have been used to prepare activated carbon [30-35]. Many raw materials have been employed to manufacture diverse materials due to their lignocellulosic biomass [36-38]. The *Robinia pseudoacacia* L. (Fabaceae) was preferred because it is widely spread in warm climates and the wet forests zone of Anatolia, Türkiye. This plant's fresh form is beneficial for many disorders since it contains polyholosides, phenolic, nitrogenous, sesquiterpenes, steroids, lipids, and minerals [39]. Also, this plant is abundant in the environment as a lignocellulosic waste.

The purpose of the paper was to use activated carbon from *Robinia pseudoacacia* L. biomass as lignocellulosic waste to eliminate gas-phase toluene with various concentrations. The produced activated carbons were also determined physicochemical properties. The removal efficiency of different concentrations of toluene, which is commonly found in the outdoor and indoor environment, with carbon-based adsorbent was investigated.

2. MATERIAL AND METHOD

2.1. Materials

All used chemicals were analytical grade, potassium hydroxide (KOH from Merck, Germany), toluene (C₇H₈, >99.9% from Sigma-Aldrich), BTEX standard solution (Ultra Scientific brand Aromatic Hydrocarbons Mixture DWM-550-1). The chemicals used in the study were used without any purification process and without allowing them to deteriorate in their pure form. The precise weighing of the samples was accomplished by Adam PW 21, England.

2.2. Production of the KN_xAC

The stems of *Robinia pseudoacacia* L. were used as a precursor for activated carbon. It was collected from Kastamonu, Türkiye, in September 2021 and dried for two months at room temperature until not damp. After the drying process, these plants were cut into very small particles (range of 1-4 cm). They were sent to ball-milling and sieved for 63-250 µm fine powder. KOH impregnated all samples in the ratio of 0.5:1, 1:1, 2:1, 3:1, and 4:1 w/w for 24 h at room temperature. They were washed with deionized water until pH was neutral to remove any remaining chemicals.

The neutral samples were dried at 105°C overnight and stored in a desiccator for characterization. The temperature ranges studied in the carbonization process are between 500-900 °C for 1.5 h under nitrogen gas (N₂) using a reactor which was put raw material ($W_{\text{raw material}}$) in high-temperature resistant steel material. The produced (W_{produced}) activated carbons were coded in Table 1 and calculated its yield percentage in Equation (1).

$$\text{Yield of AC} = \left(\frac{W_{\text{produced}}}{W_{\text{raw material}}} \right) \times 100 \quad (1)$$

Table 1 Identifications of KNACs

Sample ID	Impregnation ratio wt%	Activation temperature °C
KN ₁ AC	0.5:1	500
KN ₂ AC	0.5:1	600
KN ₃ AC	0.5:1	700
KN ₄ AC	0.5:1	800
KN ₅ AC	0.5:1	900
KN ₆ AC	1:1	500
KN ₇ AC	1:1	600
KN ₈ AC	1:1	700
KN ₉ AC	1:1	800
KN ₁₀ AC	1:1	900
KN ₁₁ AC	2:1	500
KN ₁₂ AC	2:1	600
KN ₁₃ AC	2:1	700
KN ₁₄ AC	2:1	800
KN ₁₅ AC	2:1	900
KN ₁₇ AC	3:1	600
KN ₁₈ AC	3:1	700
KN ₁₉ AC	3:1	800
KN ₂₀ AC	3:1	900
KN ₂₁ AC	4:1	500
KN ₂₂ AC	4:1	600
KN ₂₃ AC	4:1	700
KN ₂₄ AC	4:1	800
KN ₂₅ AC	4:1	900

2.3. Characterization of KNAC

The KN_xAC samples were taken from the desiccator. They carried out surface area and pore size distribution by the Brunauer–Emmett–Teller (BET) method using Quantachrome Autosorb automated gas sorption analyzer (NOVA 2200e). All characterization processes were applied to KN_xAC samples and raw material.

2.4. Gas-phase Toluene Adsorption Experiment

The experimental setup for gas-phase toluene adsorption was operated with KN_xACs. Air vacuum pump widely used in gas experiments (SKC The AirChek XR5000), a gas flow meter for a flow rate adjustment, Tenax TA sorbent in tubes for accumulation of toluene, and a reactor from quartz reactor, which the filled KN_xACs. Before the experiments, the system with different volumes was run uninterruptedly to determine the initial conditions (C_0). These experiments were made under steady humidity and temperature

conditions as 20% RH and 25 °C, respectively. It was operated with 10, 50, 100, 150, 300, 600, 800 and 1000 ppm for 30 – 60 - 90 - 120 - 150 - 180 - 210 - 240 min. Adsorption samples were collected, sealed, transported, and placed according to the US. Environmental Protection Agency Method TO-17 [40]. The percentage of toluene removal was calculated by Equation 2 which C_0 is the initial concentration, C_e is the equilibrium concentration (ppm).

$$\text{Removal \%} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

2.5. TD-GC-MS Analysis

The gas-phase toluene concentration was measured using thermal desorption-gas chromatography-mass spectrometry (TD-GC/MS). It is the addition of gas chromatography, which provides desorption of toluene heated by an inert carrier gas stream and injection or transfer to a carrier with selected ion monitoring (SIM) mode. Agilent 6890N Network Gas Chromatograph and Agilent 5975 Series Markes Unity-2 TD were used in experimental apparatus with Tenax TA sorbent tube. The calibration is based on the seven standards as 10, 20, 40, 60, 80, 100, and 200 ng/μl for 1 μl of each concentration by TD-GC/MS to establish the calibration curves. The gas column was a DB-1 series J&W 123-1063 (60 m × 0.32 mm ID × 1.0 μm, Agilent Technologies, Santa Clara, CA, USA). The injected sample volume was 1 μl the detector temperature was operated at 250°C. 40 ml/min hydrogen (H₂) gas and 20 psi pure helium (He) gas. The utilized temperature program began at 35°C min and then increased to 300°C with a rate of 5°C/min for 30 min. Each sampling tube was consecutively used with three sorbent tubes for the average value.

3. RESULTS

According to the American Society for Testing and Materials (ASTM), *Robinia pseudoacacia* L. as raw material made volatile matter, moisture,

and ash analysis by standards E872-82, E871-82 D1102-84 [41-43].

3.1. Yield of KN_xAC

One of the first targets aimed at producing activated carbon is to obtain as much activated carbon as possible from the raw material. Therefore, two factors to be considered during the production phase are the pyrolysis time and the carbonization temperature. These effects can keep the high or low product of KN_xACs. The activated carbon production performance at 500 °C was found at 39.78%, while carbonization temperature increased to 900 °C was obtained at 21.45% after the process result.

3.2. Characterization of KN_xAC

High porosity and BET surface area are required for a successfully activated carbon, measured with NOVA Touch LX4 nitrogen adsorption-desorption analysis (Quantachrome Instruments, South San Francisco, CA, USA). Also, KN_xACs were coded from KN₁₋₂₅AC according to the several impregnation ratios of KOH. The cellulose, hemicellulose, and lignin content of the biomass was found in Table 2.

Table 2 Cellulose, Hemicellulose, and Lignin content (wt%)

Cellulose wt%	Hemicellulose wt%	Lignin wt%
25.2	31.3	30.9

The composition of *Robinia pseudoacacia* L. biomass is given in Table 3.

Table 3 Composition of *Robinia pseudoacacia* L. biomass

Ash %	Moisture %	Vol atile %	Fix C %	C %	H %	N %	O %
1.3	11.88	82.7	4.2	36.2	9.1	6.5	34.9
			8	1	3	5	3

The highest surface area was obtained with 4:1 KOH at 800 °C coded as KN₂₄AC (1271.3 m²/g). Also, KN₁₉AC, KN₂₀AC, and KN₂₅AC were found in high surface areas of 1142.7, 1067.5, and

1202.8 m²/g, respectively. They were chosen for micro and total pore volume in Table 4.

Table 4 The pore structure of KN_xAC.

Samples	S _{BET} (m ² /g)	V _{micro} (cm ³ /g)	V _{total} (cm ³ /g)
KN ₁₉ AC	1142.7	0.151	0.355
KN ₂₀ AC	1067.5	0.122	0.274
KN ₂₄ AC	1271.3	0.303	0.377
KN ₂₅ AC	1202.8	0.281	0.317

The surface morphology of *Robinia pseudoacacia* L. biomass and KN₂₄AC were present with solid residue and chemicals by SEM. Their surface area was calculated by the BET method [44]. As the carbonization process temperature increases, other compounds in the activated carbon structure are eliminated because of the pore formation. However, it is seen that the disinformation in the form becomes after a specific temperature in Figure 1.

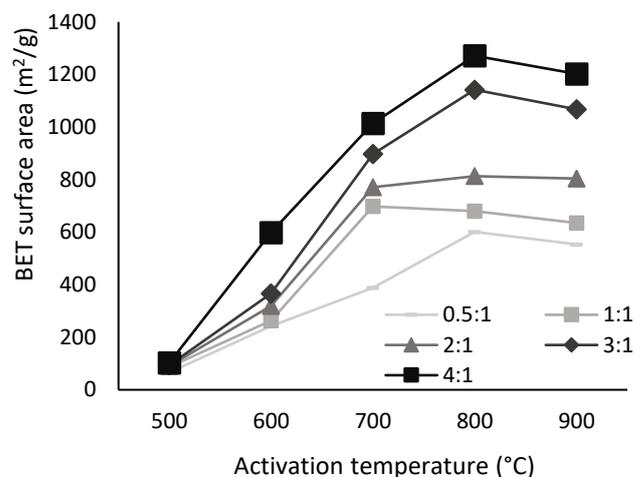
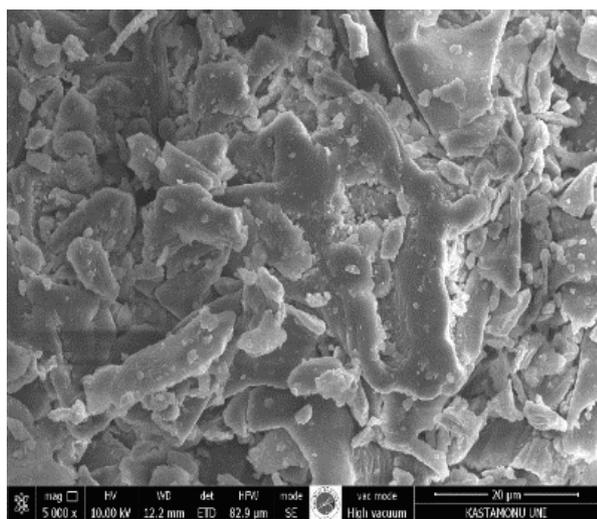
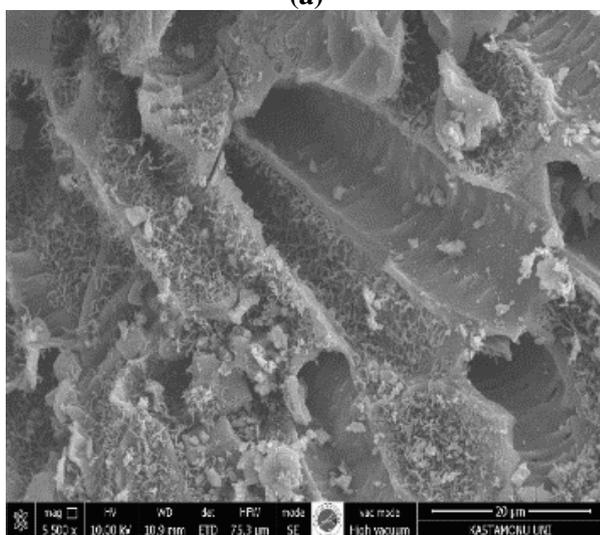


Figure 1 S_{BET} values of KN₂₄AC

Scanning Electron Microscopy (SEM) graphs were described in the *Robinia pseudoacacia* L. biomass and KN₂₄AC. The striking point is the existence of irregular structures and inhomogeneous order in the SEM microimages. The surface of raw material is smooth and has no porosity, although after the activating process has been seen. Also, their pore size has increased and become more regular in Figure 2.



(a)



(b)

Figure 2 SEM micrographs at 20 nm (a: Raw biomass, b: KN₂₄AC)

3.3. Adsorption of Toluene

According to the IUPAC classification, the adsorption behavior of KN₂₄AC was analyzed by N₂ adsorption-desorption analysis that exhibited type IV isotherm at 77 K. It indicates the presence of mesopores sizes between 2 and 50 nm or 20 and 500 Å. To determine the initial conditions of the experimental setup was operated multiple times without adding activated carbon to the system. There are several reasons for this, the most important of which is that toluene molecules are high-speed and active. It was run empty because the desire to cling to the wall and pipes of the reactor was high. The efficiency of adsorbents varies depending on initial concentration and contact time. The adsorption capacity of an

adsorbent is the milligrams of adsorbate per 1 gram of KN_xACs. The highest surface area was obtained with KN₂₄AC, although KN₁₉AC, KN₂₀AC, and KN₂₅AC were used in the toluene capture test. Before the test conditions, it was waited for about 30 minutes for the system to steady-state conditions.

3.3.1. Initial Concentration

The initial concentrations used in the study were determined as 10, 50, 100, 150, 300, 600, 800, and 1000 ppm, which are levels that can be commonly found indoors. At test conditions with 10 ppm was 4 mg/g, 111 mg/g was obtained at 1000 ppm. There is a difference in saturation value of activated carbon at low concentration (10 ppm) and high concentration (1000 ppm) in Figure 3.

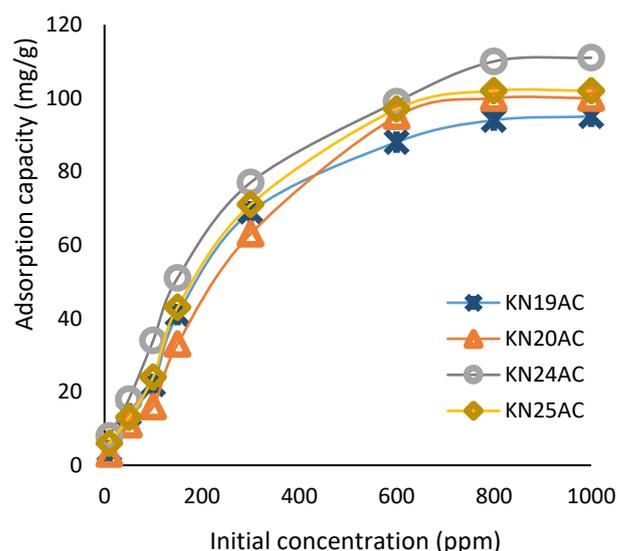


Figure 3 The plots of initial concentration gas-phase toluene according to types of KN_xACs

3.3.2. Experimental Time

The effect of contact time was examined until 240 min on the toluene adsorption study. Each measurement was made at 30-minute intervals. The retention time of toluene gas in activated carbon was fast until the 120th minute. Then, it showed a decreasing trend until the 180th minute and reached the saturation value between 180-240 in Figure 4.

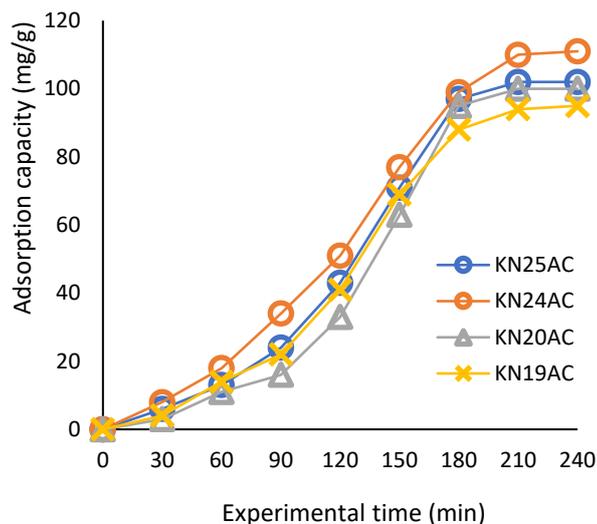


Figure 4 Variation of toluene adsorption capacity for experimental time

The binding rate, shape, and desire of toluene are directly affected by the arrangement of its molecules. The adsorbate speed was affected by plugged pores, so the bond of speed toluene molecules was decreased during the adsorption. As the pores fill, the toluene molecules need more time. The maximum adsorption capacity of gas-phase toluene was reached 111 mg/g in 1000 ppm for 240 min.

4. DISCUSSION

The performance of KN_xACs was executed with several initial concentrations between 10 and 1000 ppm at room temperature. Raw material impregnated with KOH and carbonized at 800 °C. The holding capacity of toluene molecules is higher in experiments due to the BET surface area of KN₂₄AC being highly increased. The KN₂₄AC was compared to other studies that the surface area seems tolerable for a lignocellulosic-based activated carbon [45]. Freitas et al. [37] were produced activated carbon derived from coconut shells (S_{BET} : 560 m²/g, V_{micro} : 0.19 cm³/g) and were exhibited surface areas. Also, they were analyzed commercial activated carbon and compared it with produced activated carbon (S_{BET} : 799 m²/g, V_{micro} : 0.24 cm³/g). Chung et al. [46] were used lignocellulosic waste materials such as *Acacia mangium* wood sawdust, coconut shell, sugarcane bagasse, and lignin-rich (S_{BET} : 307.92-937.43 m²/g). They were analyzed

through BET, EDS, TPD-NH₃, SEM, and FTIR. According to this study, the KN₂₄AC was demonstrated high surface area and micropore volume as 1271.3 m²/g and 0.303 cm³/g, respectively. Various pollutants were also removed with the produced activated carbons [47]. Adsorption capacities varied according to pollutant type and ambient conditions. Andrade et al. [48] were investigated VOC removal by activated carbon from mango fruit shells. The adsorption capacity was found at 472 mg/g at 30 °C and 363 mg/g at 50 °C for acetone gaseous. Shen and Zhang [29] were prepared with N-doped porous carbon from co-hydrothermal carbonization (co-HTC) of lignocellulose via KOH at 700 °C. It has 1396.5 m²/g, and an adsorption capacity was 394.2 mg/g for toluene removal. Anjum et al. [49] were modified activated carbon with benign oxidizing agents and were searched for physicochemical properties for BTX removal in aqueous. The ACs were used 0.01-1 g, different temperature ranges (25-45 °C). They found contact time, adsorbent amount, initial concentration, temperature, and pH important factors for removing benzene as 260.78 mg/g. Vikrant et al. [50] used a metal-organic framework and referenced activated carbon to compare adsorption capacity. They were reported that the high degree of M199 was 94.8 mg/g, although activated carbon was reached 93.5 mg/g in the treatment of gaseous pollutants. He et al. [51] were produced with lignin-based pitch extracted from black liquor and were found to have a high surface area of 3652 m²/g. This high-powered activated carbon was obtained at 1244.9 mg/g to capture gaseous benzene under 5000 ppm.

Generally, the variety and duration of chemical activation have been changed for high surface area in studies. The carbon and lignin contents of biomass were effectively used. Also, different molecular sizes and affinities were effective in toluene, which is desired to be removed. Adsorption studies were performed in a batch, continuous, and sequential reactor. However, these studies were tested in simultaneous high concentration indoor air. It is seen that the adsorbed amount decreases as it increases with reactor size. Thus, the material and experimental

setup are important for adsorption capacity and behavior.

5. CONCLUSION

The lignocellulosic biomass can be used as the raw material for activated carbon preparation. It is a valuable material for the removal of toluene from indoor air. Also, it is a substantial treatment in air pollution control. The lignocellulosic biomass can be regarded as ample agricultural or woody waste. An effort was made to obtain the optimum surface and micropore volume with the carbonization at the optimum temperature. It seems more appropriate to investigate the evaluation of optimum conditions with different conditions and activation agents. The carbon content of the raw material is an important factor in the production of activated carbon. In this study, the highest yield of activated carbon was carbonization at 800 °C and 90 min via KOH (4:1), resulting in activated carbon with a maximum total specific surface area of 1271.3 m²/g. Temperature value has increased the adsorbent's performance through some groups (such as hydroxyl, ethers, and aromatic compounds). Toluene is an air pollutant released from various sources, deteriorating indoor air quality. It can be carcinogenic for human health when it is excessive. Toluene removal is a very significant issue that has been widely used in various industries and industries. The aim of the study was successfully performed with KN₂₄AC, which was used as a filter for toluene removal from indoor air. As a result of the success of KNAC produced in the study in toluene gas, which is among the volatile compounds, its application to other volatile compounds is seen as a very reasonable and inexpensive method. Investigating the total removal of other VOCs on an industrial scale and conducting kinetic studies will be a pioneer for other researchers.

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The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the authors.

Authors' Contribution

The authors contributed equally to the study

The Declaration of Ethics Committee Approval

This study does not require ethics committee permission or any special permission"

The Declaration of Research and Publication Ethics

The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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