



New Method for Producing Carbon Sphere from Waste Tyre (NEWCSWT)

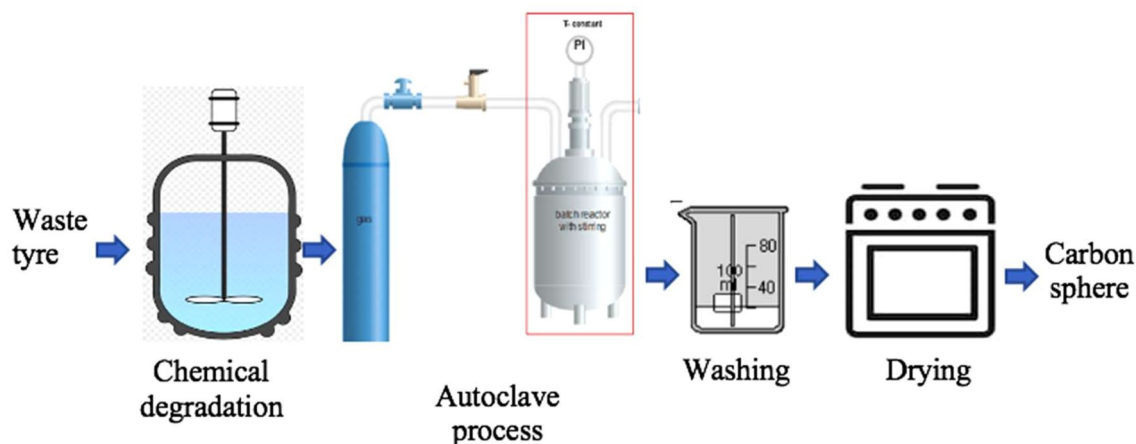
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Abstract

In the study, it was aimed to define a new method for the production of clustered and hierarchical carbon spheres from waste tyres, 80% of which cannot be used in the world. A clustered and hierarchical carbon sphere was produced by applying chemical degradation and autoclave processes to waste tyre scraps, respectively. Waste tyre slurry was obtained by degrading the waste tyre scraps after waste tyre scraps swelled in H_2SO_4 and $CH_3OH-NaOH$ solution were mixed for 15 min at room condition. Different pressure (1–2.5–5 MPa), time (2–3–4 h) and temperature (200–300–400 °C) were chosen as parameters for the production of carbon spheres from 20 g of waste tyre slurry in the autoclave process. The produced carbon spheres were characterized by CHNS, FT-IR, SEM, BET, XRD, TEM, Raman analyzes. It has been determined that the most suitable conditions for the production of clustered and hierarchical carbon spheres from waste tyre were for 3 h at 300 °C under 5 MPa. The surface area of the clustered and hierarchical carbon sphere obtained under these conditions was 559.5 m²/g. As a result, a simple and cost-effective new method was introduced using waste tyre as the carbon source for the production of carbon spheres.

Graphical Abstract



Keywords Waste tyre · Carbon sphere · Chemical degradation · Recycled carbon · Recycling · Circular economy

Statement of Novelty

In the study, it was aimed to define a new method for the production of clustered and hierarchical carbon spheres from waste tyres, 80% of which cannot be used in the world. A clustered and hierarchical carbon sphere was produced by

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applying chemical degradation and autoclave processes to waste tyre scraps, respectively. In the study, clustered and hierarchical carbon spheres were obtained from 20 g of waste tyre slurry produced as a result of chemical degradation at 50 Mpa pressure, 300 °C temperature and 3 hours. The surface area of the clustered and hierarchical carbon sphere obtained under these conditions was 559.5 m²/g. As a result, a simple and cost-effective new method was introduced using waste tyre as the carbon source for the production of carbon spheres. Limited information has been found in the literature regarding the use of waste tyre as a carbon source for carbon sphere production. The study will contribute to the literature by defining a new simple and cost-effective method for carbon sphere production using waste tyre as a carbon source.

Introduction

Among carbon-based materials, carbon spheres are very interesting due to their morphology and pore structure, as well as the ability to control their size [1]. Carbon spheres, ranging in size from nanometers to micrometers, have attracted great attention over the past decade for their remarkable role in some applications [2]. Carbon spheres (CSs) can provide conductivity, high purity, unique structure, variable and high specific surface area, and faster molecular diffusion/transfer [3, 4]. They have great potential and are becoming increasingly important as they are promising in applications such as CO₂ capture, adsorption, separation and purification of dyes, encapsulation of biomacromolecules, conversion materials, especially as energy storage and catalyst support, fuel cells, supercapacitors and lithium batteries [1, 5, 6]. Since they are more advantageous in terms of mass diffusion and transport compared to conventional carbon materials, carbon spheres are considered to be important for their various potential applications such as drug delivery, water treatment processes, energy conversion and storage, particle template and catalyst support [6].

Carbon spheres (CSs) cluster due to the attraction of Van Der Waals forces to each other. At the same time, they tend to interlace, forming necklaces or beads-like structures, and they connect with each other. Thus, although carbon spheres are not normally discussed as separate, they often form bonded extended solid chains [6].

CSs have diameters less than 1000 nm. Classification of spheres according to their diameters; (i) well graphitized spheres (2–20 nm), (ii) less graphitized spheres (50–1000 nm) and carbon beads (> 1000 nm). The dimensions of the carbon spheres can be controlled according to the parameters of the carbon source, the applied process techniques and the type of process; (1) Carbon sources such as polystyrene, coal, asphalt, melamine formaldehyde, potato

starch [6], sucrose, glucose, lactose, starch, pectin, chitosan, alginic acid, tannins, graphene oxide, phenolic resin, resorcinol, polymers; (2) Acid applications and carbonization techniques such as hydrothermal, pyrolysis, microwave; (3) Similar process type such as reaction time and temperature, precursor concentration [1]. So far, various ways including nano casting with silica spheres as rigid templates, hydrothermal carbonization of carbohydrates, arc discharge, laser ablation (plasma method), autoclave, chemical vapor deposition (CVD), modified Stöber synthesis, soft stenciling methods, polyaromatic precursor induced by the Friedel Craft reaction, spray pyrolysis have been developed for CS [2, 5]. Due to the low efficiency of carbon spherical production using the arc discharge method, the use of the technique has been limited so far. Carbon spheres were produced at high temperature (700–1000 °C) and 3–5 kW laser power in the inert gas environment of the plasma technique. In the shock compression technique, a carbon sphere was produced under a high pressure of 57 GPa by using fullerene, an expensive carbon source. The chemical vapor deposition method, in which high temperature (~ 1000 °C) reactions occur, was applied in two different ways, catalytic and non-catalytic. In the catalytic method, reactions were carried out in the presence of catalysts. In the supercritical fluid method, carbon spheres were produced under the effect of inert gas at a temperature of 550–650 °C, 1000 bar pressure and within 6–24 h. Hydrothermal processes have great potential for the preparation of carbon particles with varying morphologies and carbon spheres were produced at 200 °C in 10 h [5].

The production of nanomaterials under low temperature conditions is still a problem. Studies are carried out at high pressures to solve this problem. In the autoclave process, carbon spheres were synthesized at 30–200 MPa pressure, 200–1000 °C temperature and 3–20 h. In these studies, the duration (> 10 h) increases as the temperature or pressure decreases [5].

The production and disposal of automobile tyres are the most influential factors in environmental damage [7]. Large quantities of waste tyres are produced, which will cause serious environmental pollution due to their large volume, durability for more than 100 years, and not self-degradation. They have a strong environmental impact due to the fact that some components or degradation products are toxic compounds (CO, dioxins, dioxin-like compounds, volatile organic compounds (VOCs), PAHs, heavy metals and others). About 1 billion tyres are disposed of each year, contributing to an estimated 4 billion unwanted waste tyres already present in landfills and stockpiles. Disposal of used tyres is a global problem and more than 50% is disposed of without any application. In 2030, the number of waste tyres will reach 1.2 billion per year [8, 9]. Such large volumes of waste tyres not only pose a risk to human health and the environment, but also represent a significant waste

of a potentially valuable and carbon-rich resource. Despite significant research and numerous technological advances, viable processes for the cost-effective recycling of waste tyres do not exist. New approaches are urgently needed as the increasing mountains of waste tyres around the world continue to outpace the gains in recycling [10].

Waste tyres are predicted to be a promising carbon precursor for the production of carbon nanospheres (CNSs) due to their high carbon content, low price and abundance. A spherical carbon sphere with an average size of 50 nm was synthesized from waste tyre powder by a chemical vapor deposition method on a ferrocene catalyst at 800–900 °C. The surface area of the carbon spheres was 65 m²/g [11].

Some of the carbon sphere production methods lead to non-porous spheres with low surface area or non-uniform particles that limit their application. However, they require activation and carbonization under CO₂ (g) after an initial hydrothermal process. Carbon sphere production reports are mainly based on expensive and impractical methods that are difficult to purify and involve multi-step processes such as stencil extraction [2]. A new approach or methodology needs to be developed to produce a low cost carbon sphere with higher strength, good pore size distribution, good surface properties and high specific BET surface area [12].

In the study, clustered and hierarchical carbon spheres were obtained from 20 g of waste tyre slurry produced as a result of chemical degradation at 50 Mpa pressure, 300 °C temperature and 3 h. Limited information has been found in the literature regarding the use of waste tyre as a carbon source for carbon sphere production. The study will contribute to the literature by defining a new simple and cost-effective method for carbon sphere production using waste tyre as a carbon source.

Material and Method

Material

The samples of waste tyre scraps (WT), about 4.0 mm in diameter and 1.02 cm in length, used in the experiments were provided from a tyre retreading facility in Turkey. The chemicals, 99% CH₃OH, solid NaOH, 96% H₂SO₄, 96% C₂H₅OH, used in the experimental studies were purchased from Tekkim.

Method

Carbon spheres were produced from waste tyre scrap as a result of the application of chemical degradation and autoclave processes, respectively (Fig. 1).

Chemical Degradation

The chemical degradation process of waste tyre scraps was applied according to Patent no: 2015/13034. Waste tyre scraps were swelled at a rate of 12.27 g H₂SO₄/g WT in concentrated H₂SO₄ during 20 min at a temperature of 140 °C. A mixture of CH₃OH-NaOH was prepared as a solvent at the rate of 0.05 g NaOH /ml CH₃OH in a separate place. The tyre scraps swelled in H₂SO₄ and CH₃OH-NaOH solution were mixed in 15 min in room condition and the waste tyre scraps were degraded. The solid (waste tyre slurry) and liquid phases were separated as the mixture formed was filtered after the degradation of the waste tyres was completed [13, 14].

Autoclave Process

Carbon spheres were produced from 20 g waste tyre slurry at different times (2–3–4 h), temperatures (200–300–400 °C)

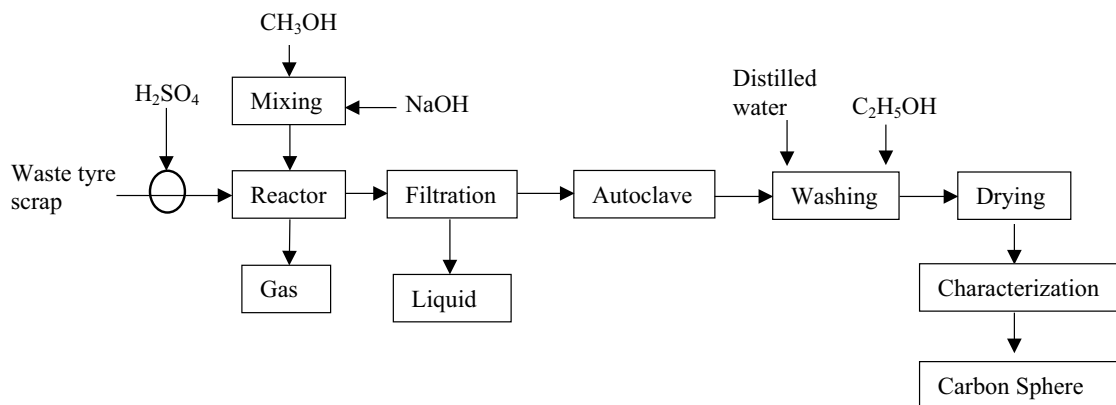


Fig. 1 Carbon sphere production process from waste tyre scrap

and pressures (1–2.5–5 MPa) under the influence of N₂(g) in high pressure and high temperature reactor (Parr 4848). Firstly, the optimum working pressure was determined at 300 °C temperature and in 2 h. Secondly, the optimum operating time at 300 °C and 5 MPa pressure, and thirdly, the most appropriate operating temperature at 5 MPa pressure and 3 h were determined. All the carbon spheres obtained were washed with ethyl alcohol and deionized water and dried at 60 °C for 8 h. Each experiment was repeated three times.

Characterization

Surface morphology (SEM- ZEISS Supra 40VP brand scanning electron microscope (1.3 nm at 15 kV) and JEOL JEM 1220 brand transmission electron microscope (100 kV, with 0.9 nm image resolution at 1 kV)), surface area (Micromeritics ASAP 2020 gas adsorption porosimeter (0.6 g sample was degasified for 24 h. at 10 °C/min. under N₂(g)), functional structure (Perkin Elmer Spectrum 100 model FT-IR spectrometer (400–4000 cm⁻¹ with KBr) and Raman Microscope-WITec alpha 300 Series (400–4000 cm⁻¹ with 532 nm and ×200 magnification at 0.5 mW (for CS) and 2 mW (for SP)), mineralogical structure (Panalytical Empyrean model HT-XRD (0.0001°/min. scan speed and from 10° to 90° for 2θ), elemental content (LECO CHNS 628) analyzes were applied to carbon sphere samples.

Results and Discussion

Carbon spheres (CS) have the potential to be used in many areas such as adsorption, separation and purification, energy storage, fuel cells, supercapacitors and batteries due to their high specific surface area and rapid molecular diffusion/transfer, and many different methods have been defined for their production [5]. In the study, production of clustered and hierarchical carbon spheres was introduced by applying autoclave process to the waste tyre slurry obtained as a result of chemical degradation.

SEM Analysis of Carbon Sphere

Carbon spheres were produced from 20 g waste tyre slurry under N₂(g) at different times (2–3–4 h), temperatures (200–300–400 °C) and pressures (1–2.5–5 MPa). The waste tyre slurry was dried at 40 °C for one night in order not to lose the volatile substances in its structure and was named Solid Product (SP). SEM views of the solid product and the produced carbon spheres are given in Fig. 2. When the surface morphologies of the carbon spheres were examined, it was observed that the spherical structures became more pronounced depending on the pressure and temperature

increase. However, it was determined that the spheres did not become clear enough after 2 h, and the structure of the formed spheres began to deteriorate after 4 h. Well-defined carbon sphere structures were obtained from 20 g of waste tyre slurry under N₂(g) at 300 °C temperature, 3 h and 5 MPa pressure. The size distribution based on SEM images of the CSs obtained under these conditions was determined to be 114 nm (Fig. 2f).

XRD Analysis of Carbon Sphere

It gives information about carbon sphere impurity, structural stress, interlayer distance and crystallinity with the help of XRD technique [5]. The interplanetary distance is calculated based on the angles of the refractive peaks. Crystallite dimensions (L_c and L_a) can be determined using angles and the full width at half maximum (FWHM) of the diffraction peaks [15]. The average crystallite is a cylinder of diameter L_a and height L_c [16].

At 21°–22°, 29° and 44° for SP were determined 10 [17], 111 [JCPDS Card No. 5-566] and 101 [18–20] peaks, respectively. The 101 and 222 peaks were detected at 44° and 82° [21] at 400 °C and 3 h, respectively. The 002 peak of the carbon sphere obtained in 5 MPa pressure, 300 °C and 3 h was determined between 26° and 27° [19, 21, ICDD-PDF # 411,487] (Fig. 3).

In sulfur-containing carbon structures, The first (002) peak at 24.6° indicates the formation of turbostratic phase, while weaker broad peak observed at 42.3° is related to (10) plane of the phase [19–22, ICDD-PDF # 411487]. The 002 peak (turbostratic carbon) at ~26.2° at 2θ gives information about the crystallinity of the carbon sphere [5, 23]. However, it has been observed that the turbostratic carbon (002) structure deteriorates and disappears in studies performed under or over 300 °C and 3 h. The 002 peak of the X-ray diffraction patterns was analyzed to determine the structural parameters. Structural parameters (d₀₀₂, L_c) were calculated the full width at half maximum (FWHM) and 2θ of the peak by using the Bragg and Scherrer equations [15, 16].

Calculation of interlayer d-spacing (d₀₀₂) using the Bragg equation;

$$d = \frac{n \times \lambda}{2 \times \sin\theta}$$

where n: positive integer; λ: wavelength; Calculation of crystal size (L_c) using the Scherrer equation:

$$L = \frac{K * \lambda}{\beta * \cos\theta}$$

where L: mean size of ordered domains; K: shape parameter (a constant); λ: X-ray wavelength; β: FWHM (in radians).

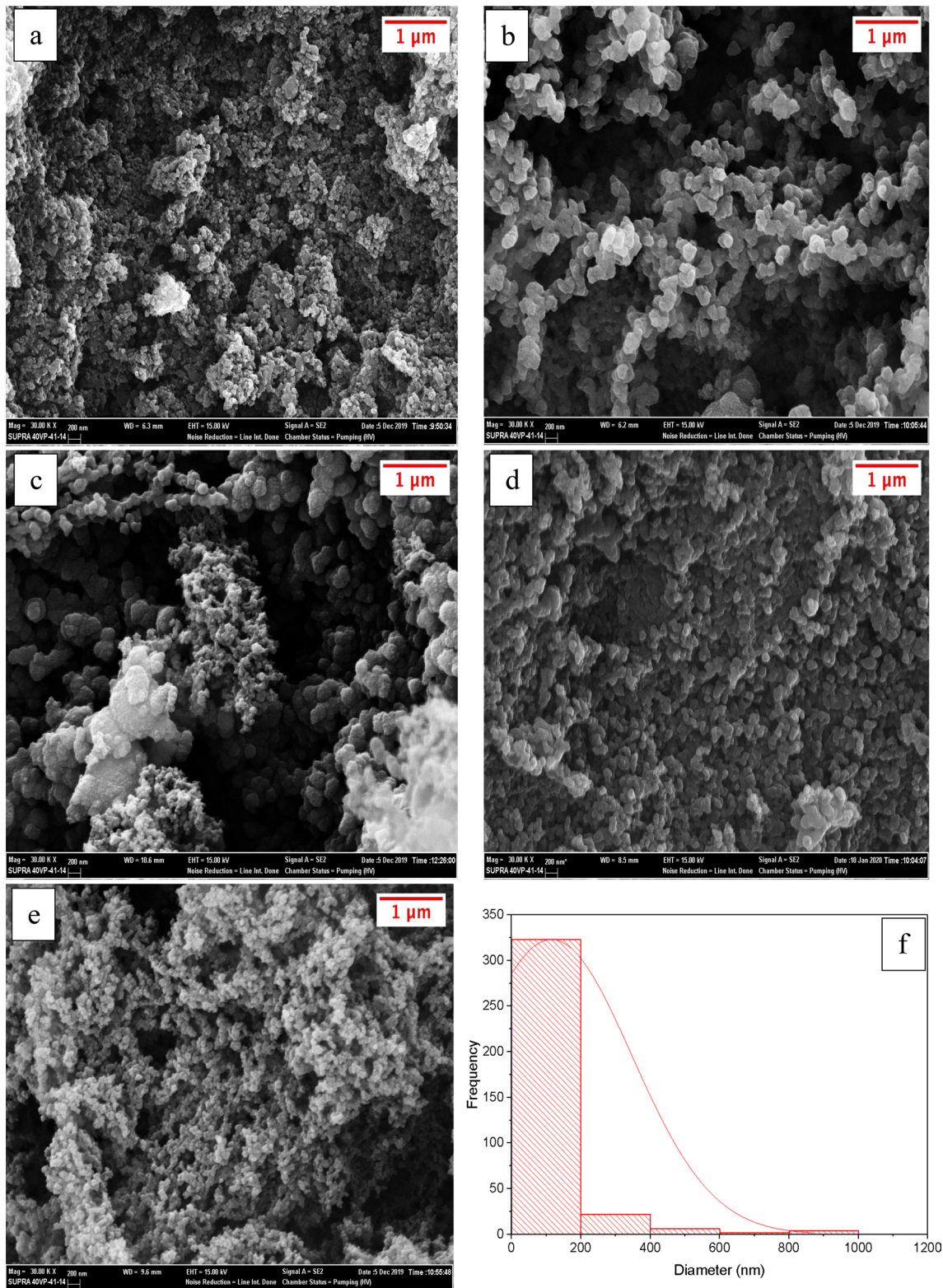


Fig. 2 SEM images of solid product and produced carbon spheres (30,000 X) (a Solid product, b 1 MPa-2 h-300 °C, c 5 MPa-2 h-300 °C, d 5 MPa-3 h-200 °C, e 5 MPa-3 h-300 °C, f Size distribution of CS obtained at 5 MPa-3 h-300 °C)

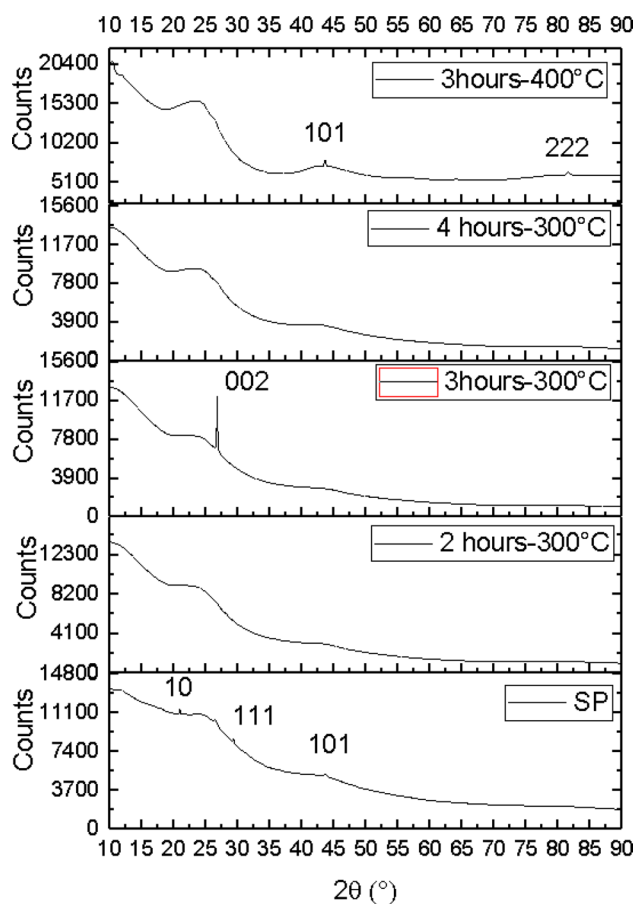


Fig. 3 XRD spectra of solid product and produced carbon spheres (5 MPa for CS)

Table 1 Elemental analysis results for WT, CS and SP

	C(%)	H(%)	N(%)	O*(%)	S(%)
WT [13]	84.77	8.07	1.88	3.96	1.32
SP	60.32	4.98	2.59	26.93	5.18
CS	66.75	3.98	2.2	22.56	4.51

*Calculated from the weight difference

The 002 peak at $\sim 26.83^\circ$ at 2θ was calculated as $\text{FWHM} = 0.14963^\circ$, $L_c = 5621.3 \text{ \AA}$ (562.13 nm) and $d_{002} = 3.32 \text{ \AA}$. L_c and d_{002} values of carbon spheres synthesized from waste tire at 1550°C for 5 s–20 min are 2.7–4.7 nm, 3.6–3.5 Å , respectively [24]. When the d_{002} and L_c values of the 002 peak of this study and the obtained carbon sphere are compared, the d_{002} values are approximately the same, but the crystal size (L_c) of the carbon sphere obtained in our study is bigger.

Elemental Analysis of Carbon Sphere

The elemental composition of the waste tire used in the study, the solid product obtained as a result of chemical degradation, and the carbon spheres are given in Table 1. Elemental composition for waste tire is 84.77% C, 8.07% H, 1.88% N, 3.96% O and 1.32% S. Nitrogen and sulfur in the structure of the waste tire result from additives and crosslinker for vulcanization, respectively [13]. The elemental composition of SP is 60.32% C, 4.98% H, 2.59% N, 26.93% O and 5.18% S. The elemental composition of CS is 66.75% C, 3.98% H, 2.2% N, 22.56% O, and 4.51% S. The sulfur in the structure of CS originates from the sulfuric acid used in the chemical decomposition process. C/H ratio of CS is 0.34.

CS obtained as a result of the hydrothermal process of glucose is 73.93% C, 4.25% H, 21.81% O. The C/H ratio of CS is 0.351–0.221 [25]. According to the synthesis conditions of carbon spheres produced as a result of sonication of water and toluene; C 73.93%, H 5.82%, N 0.65%, O 20.48% under aerobic conditions and C 75.49%, H 5.80%, N 0.39%, O 18.36% under anaerobic conditions [26]. Elemental composition of CS produced as a result of hydrothermal carbonization of enzymatic lignin hydrolysis is 88.1% C, 1.5% H, 0.2% N and 10.4% O [27]. The elemental composition of CS produced as a result of hydrothermal treatment at 190°C using sucrose as a carbon source and citric acid as a catalyst is 61% C, 5% H and 33% O [28]. The elemental composition of the carbon spheres varies depending on the carbon source used and the process applied.

Surface Area of Carbon Sphere

BET surface area is one of the most important structural features of carbon spheres [11]. It was observed that the surface area values of the obtained carbon spheres change significantly with temperature as compared to pressure and time, but the surface area values of the carbon spheres obtained below or above 3 h were lower. The surface area of SP is $17.11 \text{ m}^2/\text{g}$. The surface area value of the carbon spheres obtained at 5 MPa pressure, 3 h and 300°C temperature is $559.5 \text{ m}^2/\text{g}$ (Fig. 4) (Table 2). The surface area of SP, which is obtained by applying only chemical degradation to the waste tire, is quite low. Due to the effect of the autoclave process (high pressure and high temperature) applied to the waste tire sludge, the carbon spheres have high mesoporosity.

FT-IR Analysis of Carbon Sphere

FT-IR analysis is used to determine the chemical composition of carbonaceous materials. FT-IR spectra of waste tyre scraps, solid product and carbon spheres obtained are given

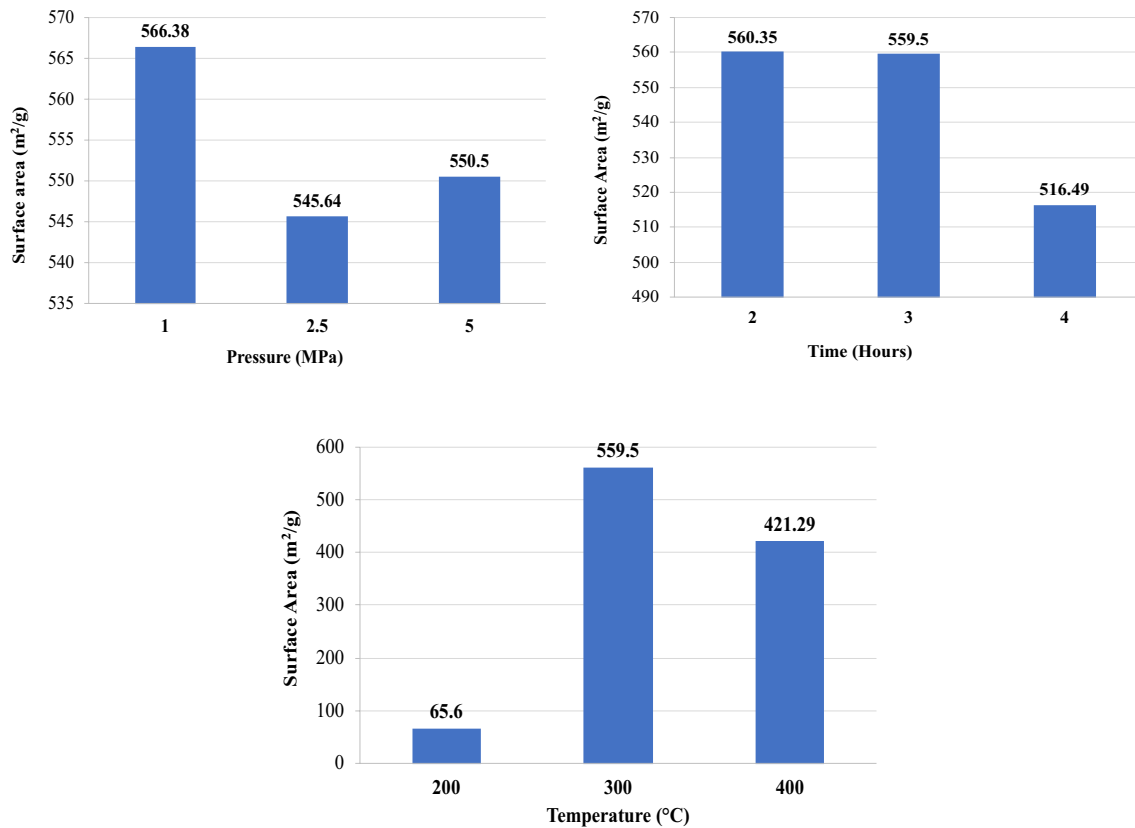


Fig. 4 Surface areas of carbon spheres obtained under different operating conditions

Table 2 Surface area comparison of solid product and carbon sphere

	S_{BET} (m ² /g)	Sext (m ² /g)	Smic (m ² /g)	Vt (cm ³ /g)	Vmic (cm ³ /g)	Vmeso (cm ³ /g)	Nanoparticle size (Å ^o)	Pore width (Å ^o)
SP	17.11	16.26	0.84	0.069	0	0.069	3506	162.66
CS	559.5	190.34	369.16	0.447	0.169	0.278	107.23	31.99

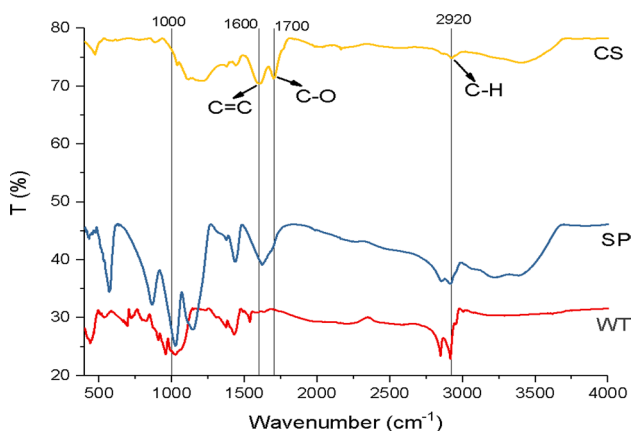


Fig. 5 FT-IR spectra of waste tyre, solid products and carbon spheres obtained at different pressures, WT waste tyre, SP solid product

in Fig. 5. The fact that the transformation to 1153 cm⁻¹ C=S and 575 cm⁻¹ S-S peaks in the solid product of 696 cm⁻¹ S-S crosslink observed in the structure of the waste tyre was proof of the chemical degradation of the waste tyre (Table 3).

~ 2900 cm⁻¹ C-H [29], 1700 cm⁻¹ C=O stretching vibration [30, 31], 1600 cm⁻¹ C=C vibrations [31, 32], 1450 cm⁻¹ C-O-C stretching [33], 1385 cm⁻¹ symmetric and asymmetric bending of CH₃ groups [34], 1200 cm⁻¹ C-O stretching [33], 1115 cm⁻¹ asymmetric stretching of C-O-C [34], 1082 cm⁻¹ C-O stretching [33], ~ 900 cm⁻¹ C-O stretching [33] peaks were observed in the structure of the carbon spheres obtained (Fig. 5).

2850–2920 cm⁻¹ C-H, ~ 1630 cm⁻¹ C=C [5] and 1730 cm⁻¹ C=O peaks seen in the FT-IR spectra of carbon

Table 3 FT-IR wavelength descriptions of SP and WT

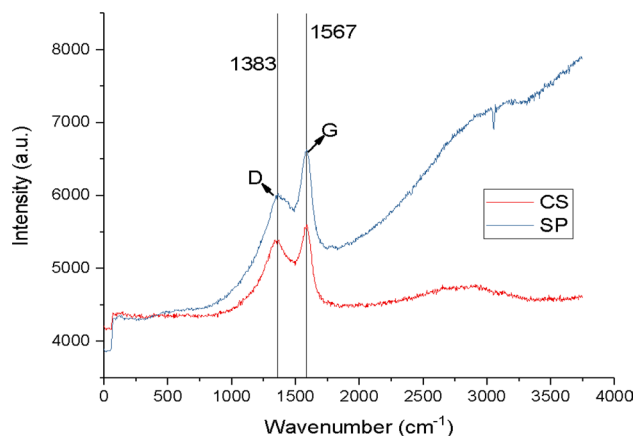
WT (cm ⁻¹)	SP (cm ⁻¹)	Descriptions
–	3400	O–H stretching absorption bands [30]
~2900	~2900	C–H stretching of CH ₃ group [29]
~2800	~2800	C–H stretching of CH ₂ group [29]
–	1630	C=C vibrations [32]
1540	–	C=C vibrations [32]
1431	1441	C–O–C stretching [33]
–	1153	C=S vibration peak [35]
1040	1030	C–O stretching [33]
959	–	C–O stretching [33]
827	867	Symmetric vibration of C–H [34]
696	–	The C–S vibration peak of the C–S–C link included in the structure C α –C β –S–S–C α' –C β' [36]
–	575	The S–S vibration peak [36]

spheres [1] were determined in FT-IR spectra of the carbon spheres obtained.

TEM Analysis of Carbon Spheres

The higher surface area of the carbon spheres produced from waste tyre by the CVD method was observed due to the decreased heating temperature and increased residence time [11]. In our study, clustered and hierarchical carbon spheres were obtained by applying for 3 h at 300 °C temperature at 5 MPa pressure under N₂ (g). TEM analysis was performed on the carbon sphere obtained under these conditions.

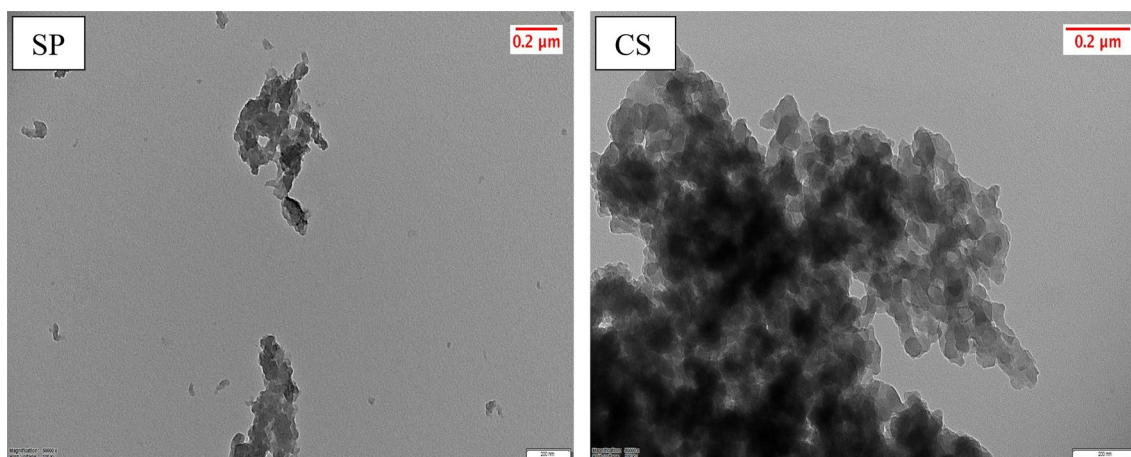
Researchers have observed that the carbon spheres produced using different raw materials have a hierarchical and clustered structure due to their intertwining and sticking

**Fig. 7** Raman spectrum of SP and CS

to each other [37–40]. It was determined that the carbon spheres obtained were clustered by intertwining according to the TEM views (Fig. 6) and that they were in a hierarchical structure according to the SEM views (Fig. 2).

Raman Analysis of Carbon Sphere

Raman spectroscopy is a very effective way to investigate the detailed bond structure of carbon films. It is used to assess the number and quality of layers by providing rapid, highly efficient and non-destructive identification of carbon layers. It is a widely used method to distinguish bond type, area size and susceptibility to internal stress in amorphous and nanocrystalline carbon films. Raman spectra are often discussed in the context of diamond versus graphite, as carbon films consist of short-range sequential sp³ and sp² bonds [41–43]. SP is a softer amorphous carbon film with high hydrogen content [41–43] (Fig. 7).

**Fig. 6** TEM images of the CS obtained at 5 MPa-3 h-300 °C and SP

The presence of irregular graphite materials [44] and carbon spheres in Raman spectra indicate the two main similar peaks that diagnose the disorder in the D-band carbon structure and the diagnosis of the G-band structural order. The D band, determined at 1383 cm^{-1} , is due to double resonance processes near the K point as well as defects and irregularities in the carbon lattice [42, 45–47]. The G band detected at 1567 cm^{-1} corresponds to the E_{2g} mode of hexagonal graphite and is related to the vibration of sp^2 -hybridized carbon atoms in the carbon layer [11] (Fig. 7). The G band is larger than the D band indicates that the structure is regular. The density ratio of D-band to G-band (I_D/I_G) is usually used to characterize the defect content of carbon structure [42]. The I_D/I_G ratio for CS and SP is 0.97 and 0.92, respectively. In some sources, the I_D/I_G ratio for CS is 0.67 [48] and 0.78 [49], respectively. The I_D/I_G ratio of CS produced from waste tire is 0.97, indicating that the carbon spheres obtained have a high amorphous carbon structure. The mean distance between defects (L_D) degree of disorder is determined using the I_D/I_G severity ratio [42, 44]. The L_D for CS and SP is 11.83 nm^2 and 11.52 nm^2 , respectively.

This indicates that the carbon spheres are composed of carbon, which is consistent with the TEM and XRD results.

Comparison with Carbon Black

It was determined with the help of XRD spectrum that carbon black is a low graphite-like material [50]. When XRD analyzes were applied to carbon blacks prepared under different conditions and subjected to various heat treatments, the patterns formed were observed to consist of crystal reflections ($00\ l$) and two-dimensional lattice reflections

(hk). It was determined that the resulting structure was parallel and equidistant, but arranged randomly [51, 52]. 002, 10 and 110 peaks were obtained as a result of XRD analysis of N770 carbon black, which is widely used in tire carcass [53]. Carbon black generally exhibits 002 and 10 peaks at 25.5° and 43° , respectively, in the case of amorphous carbon. These peaks show that each carbon atom layer in the carbon black structure is incompletely stacked and carbon black is composed of turbostratic structures [53, 54]. In general, the L_c value of carbon black is between 10 \AA and 40 \AA , depending on the thermal application applied during its manufacture [51]. The raw carbon black was composed of very small crystals [18].

Carbon black grades with large primary particles above 100 nm often show poor structure and even isolated primary particles may form (Fig. 8).

Carbon black has an O–H peak of 3440 cm^{-1} , C=C stretch at 1630 cm^{-1} and C–O stretch at 1118 cm^{-1} in the FT-IR spectrum [15].

According to the results of the carbon black Raman analysis given in the literature, the D band is almost the same as the height of the G band or higher than the G band [55, 56]. The I_D/I_G ratio of nano-sized and pure carbon black is 2.66 [57].

The elemental composition of commercial carbon black is 98.7% C, 0.4% H and 0.6% S. The surface area of commercial carbon black is $37.68\text{ m}^2/\text{g}$ [58].

When the XRD, TEM, Raman (I_D/I_G), FT-IR analysis and elemental composition information about carbon black and the obtained carbon sphere data are compared, it has been revealed that carbon spheres that do not resemble carbon black are produced from the waste tyre slurry.

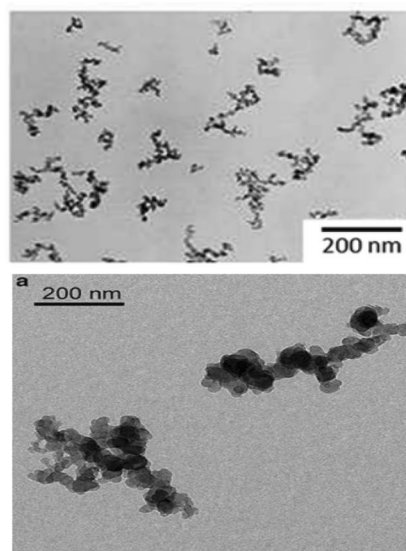
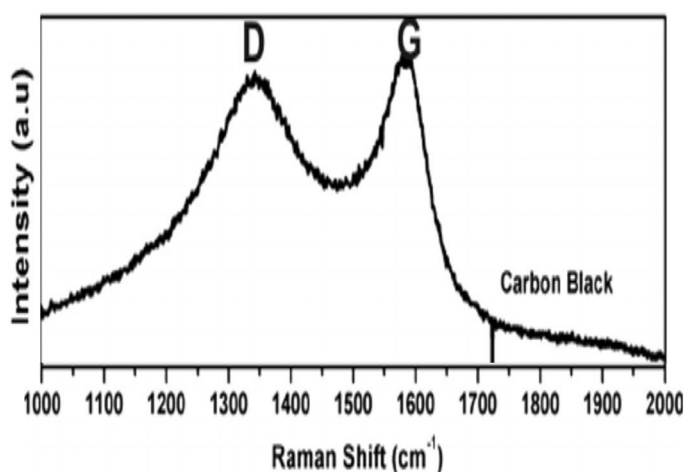


Fig. 8 TEM images [58] and Raman spectrum [53] of carbon black

Conclusion

Automotive sector continues to develop due to global developments in the world. Accordingly, the amount of waste tyres generated is increasing day by day. Only 20% of the waste tyre in the world can be used in different areas. In the study, it is aimed to describe a new method for the production of hierarchical and clustered carbon spheres from waste tyre. It has been determined that the most suitable conditions for the production of clustered and hierarchical carbon spheres from waste tyre are 3 h at 300 °C under 5 MPa pressure. The surface area of the clustered and hierarchical carbon sphere obtained under these conditions was 559.5 m²/g. 2850–2920 cm⁻¹ C–H, ~ 1630 cm⁻¹ C=C and 1730 cm⁻¹ C–O peaks seen in the FT-IR spectra of the carbon spheres were observed in the obtained hierarchical and clustered carbon spheres. The 002 peak of ~ 26.2° at 2θ gives information about the crystallinity of the carbon sphere and this peak was determined in the obtained hierarchical and clustered carbon spheres. It was determined that the carbon spheres obtained were hierarchical according to SEM analysis and clustered according to TEM analysis.

In the studies, the autoclave process conditions applied for carbon sphere production were for 3–20 h at 200–1000 °C temperature under 30–200 MPa pressure [5]. In this study, the autoclave process conditions applied for carbon sphere production were for 3 h at 300 °C temperature under 5 MPa pressure.

As a result, a simple and cost-effective new method was introduced using waste tyre as the carbon source for the production of carbon spheres. The most important advantages of the newly described method over existing techniques are energy savings and cost. According to the methods applied, energy savings were achieved by producing carbon spheres under the influence of low pressure and temperature and in a shorter time. In addition, the raw material cost was reduced by using waste tyre as a carbon source. A new solution was proposed for the production problem of carbon sphere nanomaterials under low temperature conditions.

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Data Availability The data that support the findings of this study are available from Senay Balbay but restrictions apply to the availability of these data, which were used under license for the current study, and so are not publicly available. Data are however available from the authors upon reasonable request and with permission of Senay Balbay.

Declarations

Conflict of interest The authors declare no conflicts of interest.

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