

CHEMICAL CHARACTERIZATION OF ECO-FRIENDLY RECOVERY METHOD FOR RUBBER FROM WASTE TIRES

R.J.N. AL - Jubouri, Fawzi Habeeb Jabrail*

Department of Chemistry, College of Science, University of Mosul, Mosul, Iraq

**email: fawzijabrail@uomosul.edu.iq*

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Abstract: Recycling of waste tires is the goal of many studies, where the chemical dissolution method depends on the recovery of tire rubber (TR) and carbon black (CB) from the waste tires. Petroleum ether was used to dissolve tire powder and a black solution was produced which after refluxed at 140°C for two hours with a mixture of adsorbents including silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃) and then filtered, will give clear rubber filtrate and CB precipitate and the used petroleum ether was totally regenerate. The rubber was recovered with a weight percentage of 24% (w/w), whereas CB was reclaimed with 20% (w/w) from the total waste tire used. The recovered rubber was characterized and its functional groups were studied by ¹H-NMR and FTIR spectroscopy which shows the rubber is polyisoprene blended with polybutadiene. The XRD, FESEM, and thermal analyses (TGA, DTA, and DSC) show the recovered rubber is amorphous with an elastic nature, thermally stable with endothermic behavior, and contains some inorganic impurities. On the other hand, the BET (Brunauer-Emmett-Teller) analysis, of carbon black shows it is a mesoporous material with a high specific surface area ($as = 149.44\text{m}^2 \cdot \text{g}^{-1}$) and pores with a mean diameter (13.623 nm). The particle size, structure and surface chemistry of the reclaimed CB which was measured accordingly by XRD, BET, and FESEM and it was emphasized that the CB has excellent adsorption qualities. **Keywords:** Recycling of tires, rubber, petroleum ether, carbon black, Silicon dioxide, Aluminum oxide.

1. Introduction

The large growth in the use of automobiles produced a large amount of tire consumption, creating an extreme waste problem [1]. Because of chemical cross-linking, vulcanized rubber, unlike thermoplast polymers, cannot be recycled or reprocessed. The vulcanization process can form a cross-linked structure between the chains of the rubber, which resists environmental agents, shear modulus, and temperature applications [2]. In general, tires are made of natural rubber (NR) and synthetic rubber (SR). The three-dimensional network structure after vulcanization makes tires unable to biodegrade and difficult to photochemically decompose under environmental conditions [3]. Various rubber products ranging from healthcare, military, household, construction, and automotive [4] result in a hard-to-decompose large amount of waste rubber, which causes environmental and economic problems. There are different methods for the treatment of scrap tires [5-9], but the main methods are tire retreading, heat energy utilization, rubber powder production, pyrolysis, etc. The cost of rubber powder production is high, and the requests are limited. The production of regenerated rubber is complicated and produces waste gas, causing great harm to the environment without proper treatment [10]. Heat energy utilization of scrap tires produces high calorific value and can be used, but it will cause secondary environmental pollution. Through the pyrolysis process, the waste rubber can decompose completely and produce oil, in addition to gases that also cause secondary pollution to the environment.

The chemical recycling process of rubber is considered the most common and contains organic, inorganic, and many diverse chemical processes. In the organic process, the used reagents could cause the scission of S-S or C-S bonds of the vulcanized rubber. Chemical reagents such as butyl mercaptan, thiophenols, xylene, etc. cause cleavage of sulfur cross-links at 188°C and for 4 h [11, 12]. The chemical dissolution process can reclaim rubber from waste tires using hydrocarbon solvents [13]. The solvent molecules can penetrate among the rubber chains and cause swelling.

Sometimes, the interactions that occur between solvent-polymer and polymer-polymer could encourage the rubber chains to cleave into small segments.

In the recent work, the chemical dissolution process was used mainly for the recovery of rubber and carbon black (CB) as side products from waste tires. It is noteworthy that petroleum ether was selected as a hydrocarbon reagent for rubber dissolution because of its high permeability between rubber chains; its low cost due to its availability from a high fraction of aliphatic hydrocarbon and boiling in the range of 100–140 °C, and its low toxicity and medium impact on the environment. The silicon dioxide (SiO₂) mixed with aluminum oxide (Al₂O₃) was used as a good adsorbing agent for the separation of CB from the rubber-petroleum ether solution.

2. Experimental part

2.1. Materials

End-of-life automotive tires were collected from the local market, and washed with a soft detergent and warm water. The large tire pieces are cut into small pieces and then ground into small particles of 2.0-1.5 mm. Petroleum ether (90-140°C) from ROMIL Ltd. company was purchased, silicon dioxide and aluminum oxide were received from Aldrich Chemical Company, whereas methanol was supplied from Sharlau Chemicals, which was used as received.

2.2. Rubber Recovery

The chemical dissolution process was used for the reclaim of rubber using petroleum ether for the dissolution of crushed tires. Petroleum ether 100 ml was used for dissolving 10 g of the crushed tire (10 wt %) using a 250 ml conical flask, which was closed and fixed inside the water bath at 50°C for 3 days and shaken from time to time [14].

The steeped tire solution after filtration from undissolved solid materials was transferred into a 250 ml round bottom flask. Then, to the solution, 7g of silicon dioxide and 3 g of aluminum oxide were added as adsorbing agents for adsorbing CB and isolated from the rubber solution. The mixture was heated to the boiling point of the solvent, around 140°C for two hours with gentle stirring. The hot solution was filtered, a pale yellow solution was collected, and then left to cool [15]. Methanol as a precipitator was added at 40% (V/V) to the pale yellow solution of rubber and then heated inside the water bath at 50°C. Finally, elastic material of a yellowish brown color was precipitating, and the precipitator methanol was collected and used in the next batch [15].

2.3. Carbon Black (CB) Recovery

After the hot filtration step of tire solution, the collected black precipitate in the filter paper, which is mainly CB, was collected with the undissolved solid rubber materials and treated chemically for purification in two steps, where the first was washed sequentially with different solvents, including hexane, acetone, ethanol, and methanol, by keeping the precipitate inside each solvent for 15 minutes inside a water bath at 50°C. Finally, it was washed with hot and followed with cold water, and then the precipitate CB was dried inside a vacuum oven at 100°C. In the second step, the precipitate was heated inside the tube furnace for one hour at 500°C ±10°C and then left to cool [16].

3. Results and Discussion

The chemical dissolution method was used for the recovery of rubber and CB from waste tires. The method is simple, safe, and cheap in comparison with the seriousness of pollution issues. The important point is that the method recovered both rubber and CB. Whereas, other methods, such as the incineration method or pyrolysis of the waste tires, are dangerous and have dangerous effects on the environment, they also lose rubber. Even the mechanical methods caused rubber to be lost, and the crushed tires will be used only as filling or second materials in construction supplies.

3.1. FTIR of Waste Tire

The main functional groups of the main compounds (rubber and CB) in waste tires were determined by FTIR spectroscopy using JASCO V-630 (Portland), OR 97211, U.S. in the range of

400-4000 cm^{-1} . The absorption frequencies of the fundamental functional groups of the pristine waste tire Figure 1A shows peaks at 1342 cm^{-1} and 1376 cm^{-1} for $\gamma(\text{C-H})_{\text{str}}$ of methane and methyl groups, respectively. The peaks at 1416 cm^{-1} , 2846 cm^{-1} and 2914 cm^{-1} in Figure 1A, are for $\gamma(\text{C-H})_{\text{str}}$ of the methylene group of the rubber. The peaks at 1537 cm^{-1} and 1170 cm^{-1} are for $\gamma(\text{C=O})_{\text{str}}$, $\gamma(\text{C-O})_{\text{str}}$ and $\delta(\text{O-H})_{\text{bend}}$ respectively of CB.

The band at 960 cm^{-1} in Figure 1A, is for $\gamma(\text{C=C})_{\text{str}}$ which belongs to the unsaturated bands of the rubber. FTIR wave numbers (Figure 1A) of the pristine material shows the absorption frequencies are belonging to functional groups of polyisoprene blend with polybutadiene.

3.2. Rubber and Carbon Black Recovery

Petroleum ether was used in the chemical dissolution method for the recovery of rubber from waste tires, with the help of 70% (w/w) SiO_2 and 30% (w/w) Al_2O_3 . Soaking the tire powder in a suitable solvent is important due to the compactness of the tires. Therefore, the tire powder was soaked for 72h inside petroleum ether at 50°C with gentle shaking, which afforded the potentiality for the solvent to penetrate among rubber chains [17]. The problem of the separation of CB from the rubber solution was solved by adding the adsorbing agent. Accordingly, a mixture of SiO_2 and Al_2O_3 was used as adsorbing agents for the separation of CB and it showed a strong ability for adsorbing. The method showed a recovery of 24% (w/w) rubber and 17.4% (w/w) carbon black from the total waste tire powder used. The recovered materials were characterized using different analysis techniques.

3.3 Recovered Rubber Characterization

3.3.1. FTIR spectroscopy

Recovered rubber was characterized by FTIR (Figure 1B). Where the peaks at 1452 cm^{-1} , 2852 cm^{-1} and 2921 cm^{-1} represent the $\gamma(\text{C-H})_{\text{str}}$ of the methylene group. The peaks at 1308 cm^{-1} and 1376 cm^{-1} are of $\gamma(\text{C-H})_{\text{str}}$ of methine and methyl groups respectively. The bands at 968 cm^{-1} and 1605 cm^{-1} Figure 1B, belong to $\gamma(\text{C=C})_{\text{str}}$ of alkenes groups in rubber. The peak at 719 cm^{-1} Figure 1B, is of $\gamma(\text{C-C})_{\text{str}}$ aliphatic of the rubber chains. Those peaks at 1016 cm^{-1} and 1160 cm^{-1} represent the $\delta(\text{C-H})_{\text{str}}$ aliphatic and in the plane groups of the rubber. The mentioned bands confirm their return to polyisoprene and polybutadiene rubber.

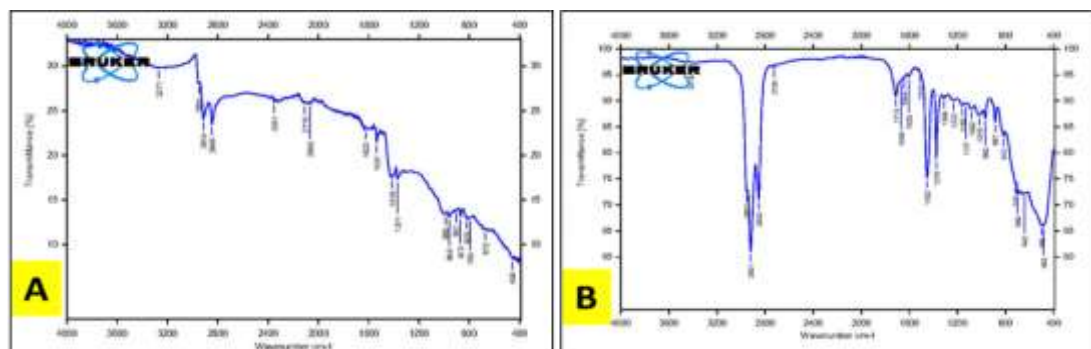


Fig. 1. FTIR spectrum of pristine (A), and waste recovered (B) rubber using petroleum ether.

3.3.2. ^1H NMR Spectroscopy

The reclaimed rubber structure was studied by ^1H NMR spectroscopy using Bruker Biospin, Avance III 500, which has variable temperature unit B-VT 3000, Germany, using deuterated chloroform solvent. The proton resonance Figure 2 shows proton resonance (m, 16H) at chemical shift $\sigma = 0.69\text{--}0.83$ ppm, which belongs to the ($-\text{CH}_3$) methyl group present in reclaimed rubber. The proton resonance (s, 77H) at 1.15 ppm belongs to the ($-\text{CH}_2-$) methylene group of the rubber.

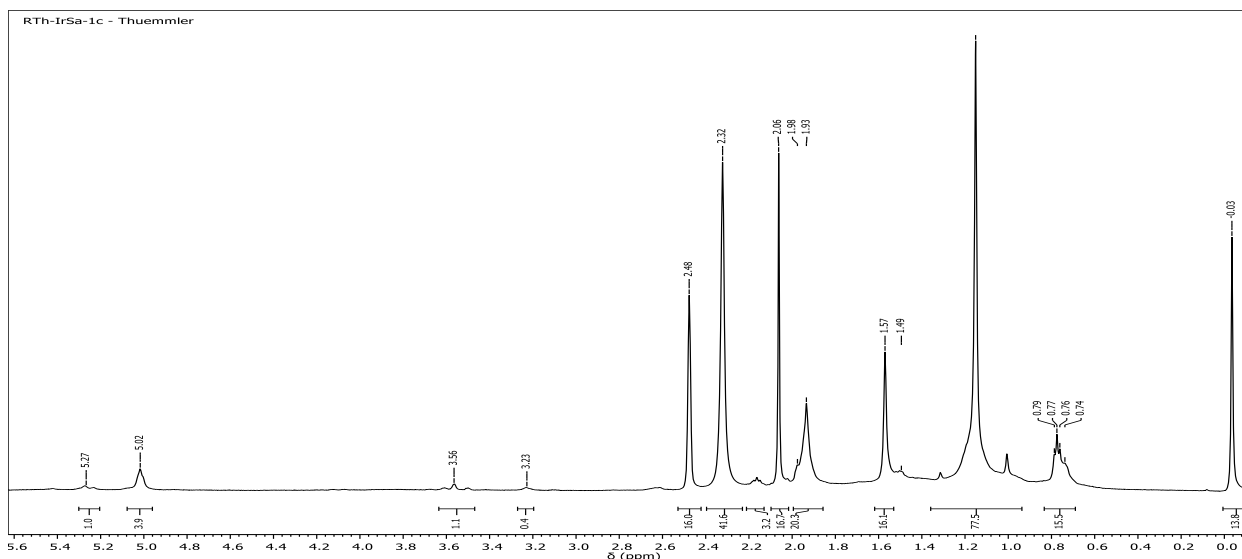


Fig. 2. $^1\text{H-NMR}$ spectrum of recovered rubber, using petroleum ether

Peak list: $^1\text{H-NMR}$ (500 MHz, CDCl_3) δ 5.27, 5.02, 3.56, 3.23, 2.48, 2.32, 2.06, 1.98, 1.93, 1.57, 1.49, 1.15, 0.79, 0.77, 0.76, 0.74, -0.03.

Multiplets [$^1\text{H-NMR}$ (500 MHz, Chloroform-d) δ 5.27 (s, 1H), 5.02 (s, 4H), 3.56 (s, 1H), 2.48 (s, 16H), 2.32 (s, 42H), 2.16 (t, $J = 7.6$ Hz, 3H), 2.06 (s, 17H), 1.95 (d, $J = 20.8$ Hz, 20H), 1.57 (s, 16H), 1.15 (s, 77H), 0.83 – 0.69 (m, 16H), -0.03 (s, 14H)].

The proton resonance (s, 16 H) Figure 2, at 1.57 is for the (=CH-) methine group of recovered rubber. The proton resonance (d, $J=20.8\text{Hz}$, 20 H) at $\sigma = 1.95$ ppm belongs to the proton which is next to a double bond i.e. an allylic group of the rubber [18]. The proton resonance (s, 4H) and (s, 1H) Figure 2, at $\sigma = 5.02$ ppm and $\sigma = 5.27$ ppm those chemical shifts belong to (-C=C-) alkene group of the reclaimed rubber. The studied protons and their chemical shifts confirm their returned to polybutadiene and polyisoprene rubber.

3.3.3. XRD Analysis

The x-ray diffraction of the recovered rubber was measured using a Philips x-ray (PW-1730) with a cu-ka radiation target with a nickel filter, with 30Kv current and 30mA voltages. The XRD pattern in Figure 3 and Table 1 shows the recovered rather has intensive peaks with a long 2θ axis, which belong to the remaining inorganic additive and some remaining adsorbing agent. Most rubbers have an amorphous nature with a low crystalline percentage.

Table 1: XRD Data of recovered rubber

Pos. [$^{\circ}2\text{Th.}$]	Height [cts]	FWHM [$^{\circ}2\text{Th.}$]	d-spacing [Å]	Rel. Int. [%]	Tip width [$^{\circ}2\text{Th.}$]
25.501	1986.52	0.2952	3.49305	100	0.3542
38.0345	536.67	0.2952	2.36591	27.02	0.3542
38.7971	171.62	0.2952	2.32114	8.64	0.3542
48.2033	755.2	0.2952	1.88790	38.02	0.3542
54.1159	514.14	0.2952	1.69477	25.88	0.3542
55.3068	555.23	0.246	1.66107	27.95	0.2952
62.854	508.89	0.1476	1.47856	25.62	0.1771
69.0264	231.65	0.1968	1.36065	11.66	0.2362
70.4675	183.63	0.2952	1.33631	9.24	0.3542
75.2178	272.78	0.3	1.26223	13.73	0.36

However, the adsorbing agent Al_2O_3 has the following intensive peaks which are similar to those found in Figure 3 and Table 1, 25.4° , 35° ; 37.7° , 43.8° , 52.5° , 57.4° , 66.5° and 68.1° [19]. In addition, the adsorbed agent SiO_2 has one broad peak and a long 2θ axis at 22.6° [20].

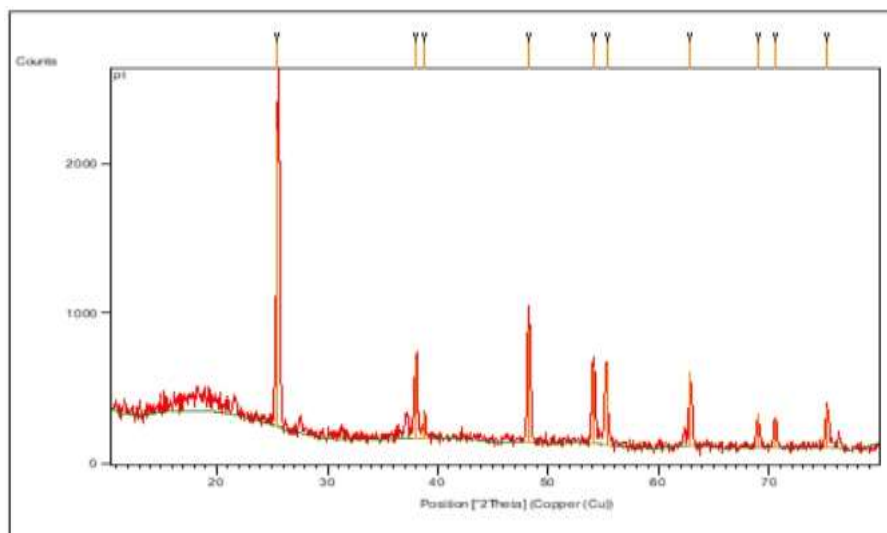


Fig. 3. XRD pattern of the rubber reclaimed by petroleum ether

The peaks of the adsorbing agent are very close to those of Figure 3, which means the rubber is not pure and is recovered with some impurities.

3.3.4. Thermal Analysis

Reclaimed rubber was studied thermally (TGA, DTA, and DSC) using TA instrument SDT Q600, UB, NY, 14260, USA. The heat rate was 80°C/min, and Al_2O_3 was the reference. The TGA thermogram Figure 4 and Table 2 shows the weight loss (%) at the initial decomposition temperature (IDT) of 92°C was 0.6% while it is 83.2% at 601°C the final decomposition temperature (FDT). At maximum decomposition temperature (T_{max}) the weight loss was 44.8% and at crystalline decomposition temperature (T_{cr}) the weight loss was 77.7%.

Table 2. Thermal data of the reclaimed rubber

Sample	TAG weight loss %				DTA °C/mg	DSC W/g	
	IDT	FDT	T_{MAX}	T_{cr}	Docomp. rate °C.min/mg	T_g °C	ΔH_f J/g
Recover rubber	0.6	83.2	44.8	77.7	0.007453	<0	+6.368
	92 °C	601 °C	358 °C	475 °C	473 °C		473 °C

The TGA data in Figure 4 and Table 2 show the recovered rubber has almost the same data as the polybutadiene [21] and polyisoprene [22].

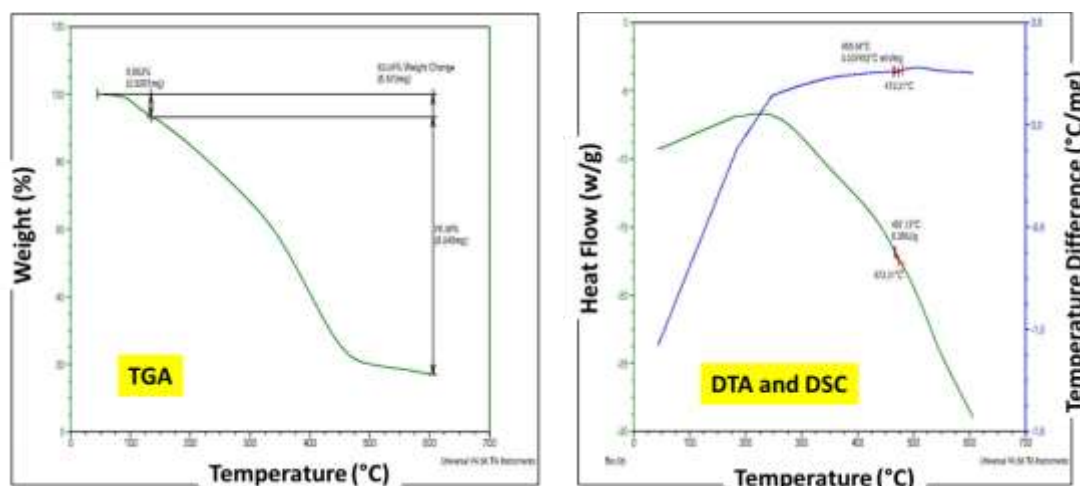


Fig. 4. TGA, DTA and DSC thermograms of rubber reclaimed by petroleum ether

The DTA thermogram of the recovered rubber in Figure 4 and Table 2, shows maximum decomposition rate of the rubber was very low $0.007453^{\circ}\text{C}\cdot\text{min}/\text{mg}$ at 473°C . Finally, the DSC thermogram (Figure 4 and Table 2) shows the heat of fusion $\Delta H_f = +6.368 \text{ J/g}$ at 473°C is an endothermic and thermally stable material and these specifications are for polybutadiene and polyisoprene rubber [23].

3.3.5. FESEM Analysis

The FESEM analysis of recovered rubber was studied using the TESCAN MIRA FESEM instrument, Brno, Czech Republic. The FESEM image (Figure 5) shows elastic surface morphology and its materials are homogeneous. The folds and ripples appear clearly on the sample surface. Moreover, the FESEM image shows the reclaimed rubber has an amorphous surface with crystal deficiency. All the aforementioned features indicate that the recovered rubber is a blend of polybutadiene and polyisoprene [24].

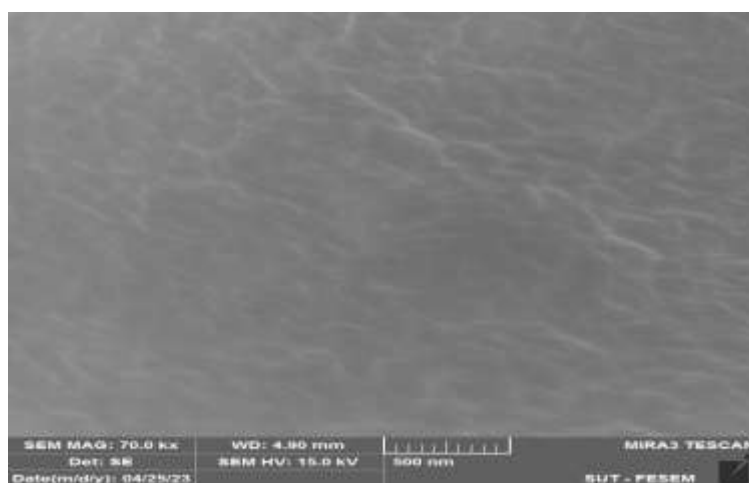


Fig. 5. FESEM image of recovered rubber

3.4. CB Recovered

CB can be recovered as by a product material from waste tire solution. Where soaking the tire waste powder in petroleum ether gives a turbid solution, but after reflux and hot filtration, it will give a clear filtrate and a black precipitate. The black precipitate beside the undissolved rubber waste in petroleum ether is mainly composed of CB, an adsorbing agent, and traces of inorganic additives. The precipitate was washed with different organic and inorganic solvents and burned inside the furnace at $500^{\circ}\text{C} \pm 10^{\circ}\text{C}$ for the removal of contaminants. Finally, the collected CB was characterized.

3.4.1. XRD Analysis.

The XRD pattern of the recovered CB (Figure 6A) shows the collected material is amorphous and has only one intense peak at 47.8° a long 2θ axis representing the graphite structure of the CB [25]. The hump-like peak at 24.0° a long 2θ axis represents the featured peak of amorphicity of CB [26]. Finally, an XRD study Figure 6A shows the recovered CB is pure material that has an amorphous nature and the individual peak was for graphite oxide present with the CB sample.

3.4.2. BET Studies

The specific surface area of the reclaimed CB particles and their pore area and other physical parameters were studied by BET measurement using surface area and porosimetry analyzer type BELSORP MINI II, Japan. The Langmuir-specific surface area is ($a_s=126.87 \text{ m}^2\cdot\text{g}^{-1}$), whereas the BET-specific surface area is ($a_s=149.44 \text{ m}^2\cdot\text{g}^{-1}$). The pore area between the CB particles from the BJH plot is ($r_p=4.61\text{nm}$) with a specific surface area of ($176.12 \text{ m}^2\cdot\text{g}^{-1}$). On the other hand, the pore volume of CB from the BET plot is ($0.5089 \text{ cm}^3 \text{ g}^{-1}$) with a pore diameter of (13.623nm). In general, the collected data from BET analysis shows the recovered CB has a high specific surface area, which means its particles are mesoporous and these are important properties for CB to become an industrial material.

3.4.3. FESEM Analysis

The FESEM image of the recovered CB (Figure 6B) shows clusters of CB particles have spherical shapes and are interspersed with numerous holes. The CB particles Figure 6B shows smooth surfaces and particles with uniform sizes.

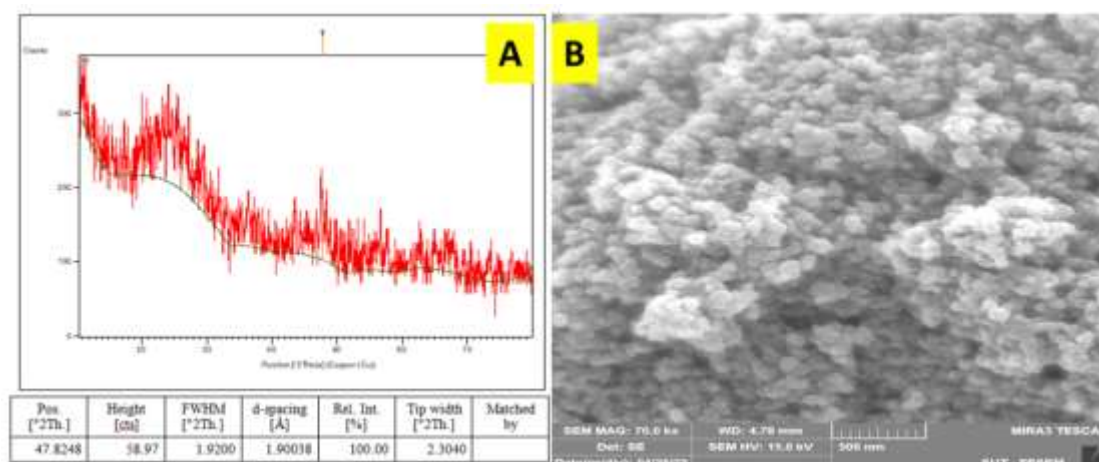


Fig. 6. XRD pattern (A) and FESEM image (B) of the recovered CB

4. Conclusions

The chemical dissolution process used for the recovery of rubber and CB from waste tires depends on petroleum ether and a mixture of active absorbing agents (SiO_2 and Al_2O_3) and shows an effective rubber recovery process from a quantitative and qualitative viewpoint. Petroleum ether, the hydrocarbon solvent, was shown to be highly effective in the reclamation of rubber due to its success in dissolving the rubber chains. Moreover, the use of 10 wt% of waste tire powder in petroleum ether and adding 70 % (w/w) SiO_2 and 30% (w/w) Al_2O_3 could reclaim 24 % (w/w) rubber and 20 % (w/w) CB from the total waste tire sample. At the same time, the used chemical process could almost completely regenerate petroleum ether, the hydrocarbon solvent beside the precipitator methanol, and be used successively in other batches.

The analyses of the recovered materials have shown that the reclaimed rubber is a mixture of polybutadiene and polyisoprene, has an amorphous nature, is not pure, but still contains some additives. Thermally, the recovered rubber is stable and has a homogeneous morphology and elastic surface. The analyses of the recovered CB contain spherical particles with an amorphous nature and a high specific surface area, besides, they are mesoporous, which are good industrial properties for CB. The previous characterizations of both reclaimed rubber and CB show they have chemical, physical, and mechanical specifications qualified for renewal of scrap tires, sheet rubber, steps, bleachers, and playgrounds. Whereas, the reclaimed CB was shown to have a good specific absorption area, and its mesoporous properties are useful for reuse in tire manufacturing.

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TULLANTI ŞİNLƏRDƏN REZİNİN TƏKRAR EMALI ÜÇÜN EKOLOJİ ÜSULUN KİMYƏVİ XÜSUSİYYƏTLƏRİ

R.J.N. AL-Cuburi, F.H. Cəbrayıl*

Kimya şöbəsi, Elm Kolleci, Mosul Universiteti, Mosul, İraq

**e-mail: fawzijabrail@uomosul.edu.iq*

Xülasə: Tullantı təkərlərin təkrar emalı bir çox tədqiqatçıların tədqiqat obyektidir. Bu tədqiqatlarda təkrar rezininin (TR) və texniki karbonun (TC) tullantı təkərlərdən bərpası kimyəvi həllolmadan asılıdır. Şin tozunu həll etmək üçün neft efirindən istifadə edilmiş və alınmış qara məhlul 140°C-də iki saat ərzində tərkibində silisium oksid (SiO₂) və alüminium oksid (Al₂O₃) olan adsorbentlərlə qaynadılmışdır. Sonra məhlul süzülüb və nəticədə şəffaf kauçukun məhlulu və TC çöküntüsü alınmışdır, istifadə edilmiş neft efiri isə tamamilə regenerasiya edilmişdir. İstifadə olunan təkərlərin ümumi miqdarına nəzərən regenerasiya edilən kauçuk kütlə faizi 24%, TC isə 20% təşkil etmişdir. Bərpa edilmiş rezin və onun funksional qrupları ¹H-NMR və İQ spektroskopiyaya üsulları ilə tədqiq olunmuş və müəyyən edilmişdir ki, o polibutadien qarışıqlı poliizoprendən ibarətdir. Rentgen difraksiya, FESEM və termiki analiz (TGA, DTA və DSC) üsullarının nəticələri göstərmişdir ki, bərpa olunan rezin elastikdir, termiki sabitdir və tərkibində bəzi qeyri-üzvi aşqarlar var. Digər tərəfdən, texniki karbonun BET (Brunauer-Emmett-Teller) analizi göstərir ki, o, yüksək xüsusi səth sahəsinə (as = 149,44 m².g⁻¹) və orta diametrə (13.623 nm) malik mezoməsaməli materialdır. Texniki karbonun hissəciklərinin ölçüsü, quruluşu və səth kimyası müvafiq olaraq rentgen difraksiya, FESEM və BET üsulları ilə tədqiq olunaraq, yüksək adsorbsiya xassələrinə malik olduğu müəyyənləşdirilmişdir.

Açar sözləri: təkərlərin təkrar emalı, rezin, neft efiri, karbon qarası, silisium 4-oksidi, alüminium oksidi.

ХИМИЧЕСКАЯ ХАРАКТЕРИСТИКА ЭКОЛОГИЧЕСКОГО МЕТОДА ПЕРЕРАБОТКИ РЕЗИНЫ ИЗ ОТХОДНЫХ ШИН

Р.Дж.Н. АЛ-Джубури, Ф.Х. Джабраил*

Кафедра химии, Научный колледж Мосульского университета, Мосул, Ирак

**e-mail: fawzijabrail@uomosul.edu.iq*

Резюме: Переработка отработанных шин является целью многих исследований, в которых метод химического растворения зависит от извлечения шинной резины (ШР) и технического углерода (ТУ) из отработанных шин. Для растворения порошка шин использовался петролейный (нефтяной) эфир, и был получен черный раствор, который после кипячения с обратным холодильником при 140°C в течение двух часов со смесью адсорбентов, включающих диоксид кремния (SiO₂) и оксид алюминия (Al₂O₃), был отфильтрован, в результате чего получили прозрачный фильтрат каучука и осадок ТУ, а использованный

петролейный эфир полностью регенерировали. Каучук был регенерирован с массовым процентом 24% (по весу), а ТУ - с массовым процентом 20% (по весу) от общего количества использованных шин. Восстановленный каучук был охарактеризован, а его функциональные группы были изучены с помощью ^1H -ЯМР и ИК-спектроскопии, которые показали, что каучук представляет собой полиизопрен, смешанный с полибутадиеном. Исследования рентгеновской дифракции, FESEM и термический анализ (ТГА, ДТА и ДСК) показали, что восстановленный каучук является аморфным, эластичным по природе, термически стабильным с эндотермическим поведением, и содержит некоторые неорганические примеси. С другой стороны, BET (Brunauer-Emmett-Teller) анализ технического углерода показывает, что это мезопористый материал с высокой удельной поверхностью ($a_s = 149,44 \text{ м}^2 \cdot \text{г}^{-1}$) и порами со средним диаметром (13,623 нм). Размер частиц, структура и химия поверхности регенерированного ТУ были измерены соответственно с помощью XRD, BET и FESEM, и было выявлено, что он обладает превосходными адсорбционными свойствами.

Ключевые слова: переработка шин, резина, петролейный эфир, технический углерод, диоксид кремния, оксид алюминия.