

## Parametric Studies on Radiation Grafting of Tripropylene Glycol Diacrylate onto Waste Tire Dust

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### ABSTRACT

Waste tire dust (WTD) was activated and modified via an electron beam to graft with tripropylene glycol diacrylate (TPGDA) monomer. This process was investigated in conjunction with reaction parameters, namely, monomer concentration, absorbed dose, grafting temperature, and grafting time. The effect of grafting conditions on the grafting yield (GY) was explored. It was found that GY increased with the increase of absorbed dose, TPGDA monomer concentration, and the grafting temperature. However, a further increase in the grafting reaction time did not affect the GY. The radiation-induced grafting technique used in this study was successful, with the maximum GY of 24.72 at an optimum grafting parameter of 5 w/v% TPGDA concentration, 60 kGy irradiation dose, 3 hours reaction time, and 60 °C reaction temperature. Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) were employed to provide evidence for the formation of graft copolymers in the grafting systems. It is evident from the FTIR analysis that the radiation grafting method successfully introduced a large number of carbonyl groups onto the WTD surface, as demonstrated by the emergence of a new peak at 1720 cm<sup>-1</sup>. The surface morphology of the grafted WTD appeared thicker, swelled, and coated compared to non-grafted WTD, followed by the integration of the TPGDA monomer into the backbone of the WTD, resulting in the expansion of the WTD's average diameter by approximately 111.5%. The results of the present study demonstrate that radiation-induced grafting techniques can be successfully employed to prepare grafted WTD.

**Keywords:** Radiation grafting, Tripropylene glycol diacrylate, Waste tire dust, Grafting parameter, Grafting yield

### 1. INTRODUCTION

In recent years, the management of discarded tires has become a significant issue. Various strategies are recommended for handling discarded tires at the end of their useful lives, including dumping, retreading, reusing, combustion for fuel, and recycling to obtain raw materials. Estimated data suggest that approximately 1.2 billion tires will be discarded annually worldwide by the end of 2030 [1], with roughly 1 billion tires becoming unfit for further use or retreading each year [2]. Improper tire management and burning can have detrimental effects on air quality, human health, and the environment [3]. To promote sustainability, the principles of the 4R's; reuse, reduce, recycle, and recovery, can be applied to regulate scrap tire disposal [4]. The waste tire products are illustrated in Figure 1 [5].

The recyclability of waste tires is challenging due to the vulcanized rubber matrix phase, which cannot be dissolved, making it incompatible with most polymeric matrices. The crosslinked structure of vulcanized rubber may impede molecular interaction and interfacial adhesion [6], leading to deterioration in both mechanical and physical characteristics [7-8]. To address this issue, extensive research has been conducted on treating waste tire dust (WTD) surfaces before and after blending processes, including methods such as in-situ polymerization, sintering of WTD, and irradiation [9-11]. High-energy irradiation

using an electron beam is considered a clean method with minimal chemical usage. According to a study by [12], pre-irradiation emulsion grafting techniques have successfully enhanced the surface free energy and hydrophilic properties of polybutadiene rubber. However, no research has yet utilized pre-irradiation emulsion grafting techniques to graft new functional groups onto WTD surfaces to enhance their properties for further blending with other polymeric materials.

For WTD to be applicable in practical settings, ensuring strong adhesion between WTD and the polymer matrix is crucial [13]. The highly cross-linked sulfur network present in waste tires renders them insoluble and infusible, making recycling into other polymeric materials challenging [11], [14-15]. Therefore, enhancing the physicochemical properties of WTD with a new monomer can improve its interaction with other polymers. Deng et al. (2020) demonstrated that tripropylene glycol diacrylate (TPGDA) exhibits high efficiency in forming a graft copolymer [17]. TPGDA's four free radical sites serve as crosslinkers, generating macroradicals that react with the polymer backbone, resulting in a well-defined three-dimensional network. Moreover, the abundance of double bonds in TPGDA increases the potential attachment sites, favoring its grafting onto WTD. While previous studies have explored the impact of TPGDA on various polymer materials [18-21], none have investigated its grafting onto WTD via radiation-induced graft copolymerization to enhance WTD's

physicochemical properties. Thus, in this study, TPGDA is utilized to graft onto the WTD rubber backbone before incorporating it into other polymeric blends. The grafted

WTD's newly developed properties have been confirmed and studied.



**Figure 1** Waste tire products. (a) Stockpile of whole waste tires, (b) Tire derived products [5].

## 2. EXPERIMENTAL

### 2.1. Materials

Waste tire dust (WTD) was sourced from Sin Rubtech Consultancy Sdn. Bhd. in Malaysia, while Tripropylene glycol diacrylate (TPGDA) was obtained from Allnex Malaysia Sdn. Bhd., also located in Malaysia. Isopropanol of industrial grade and analytical-grade methanol were procured from Fisher Scientific. All solvents were utilized as received without additional purification. Ultra-pure water (organic-free) from the purification water system was acquired from Sartorius Arium and was utilized for all sample preparations.

### 2.2. Preparation of Tripropylene Glycol Diacrylate Grafted onto Waste Tire Dust

Dried WTD was placed in a tube and positioned on a tray for irradiation using an electron beam accelerator, EPS 3000, operated in ambient air at a voltage of 2 MeV, at various absorbed radiation doses (20, 40, 60, 80, and 100 kGy). TPGDA emulsion was prepared at different monomer concentrations (3, 5, and 7 wt%) [22]. Subsequently, the irradiated WTD was immersed in TPGDA emulsions and subjected to different durations (1, 3, 5, 7, and 24 h) and temperatures (40, 50, 60, and 80 °C) to initiate the grafting process. Following the grafting process, the WTD samples were rinsed and dried in an oven until reaching a constant weight. The process of pre-irradiation emulsion grafting of TPGDA monomer onto WTD is depicted in Figure 2.

In this study, GY was utilized to quantify the relative amount of TPGDA monomer successfully grafted onto the WTD rubber backbone. This was determined by measuring the

area of the characteristic peak corresponding to the carbonyl, C=O groups at 1720  $\text{cm}^{-1}$  and calculating the ratio of this peak to the peak at 2847  $\text{cm}^{-1}$  resulting from the C-H stretching vibration of a methylene group (-CH<sub>2</sub>-), selected as the internal reference peak for WTD. For each sample, the peak area of the carbonyl bond from TPGDA is calculated from 1680  $\text{cm}^{-1}$  to 1765  $\text{cm}^{-1}$ , while the reference peak area at A2847 is calculated from 2825  $\text{cm}^{-1}$  to 2865  $\text{cm}^{-1}$  [23]. Subsequently, the grafting yield (GY) quantified from the FTIR spectra can be calculated as follows:

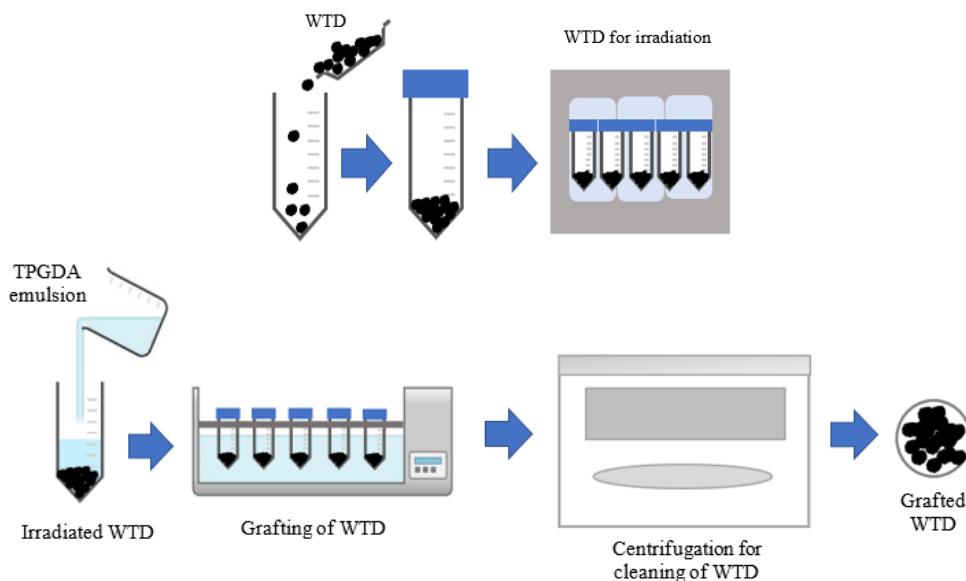
$$GY (\%) = \frac{A_{\text{characteristic peak of monomer}}}{A_{\text{reference peak of trunk polymer}}} \times 100 \quad (1)$$

where,  $A_{\text{characteristic peak of monomer}}$  is the absorbance area of the monomer,  $A_{\text{reference peak of trunk polymer}}$  is the absorbance area of the trunk polymer.

### 2.4. Physicochemical Characterization

Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) spectra were acquired using a Bruker Tensor II instrument. The samples were placed on the crystal and scanned over a range of 4000-500  $\text{cm}^{-1}$  using the OPUS software.

The surface morphology of both non-grafted and grafted WTD samples was investigated using FEI Quanta 400 Scanning Electron Microscopy (SEM). Prior to mounting on stubs, the surfaces of these samples were sputter-coated with gold using the SEM Coating Unit Biorad-Polaron Division to prevent electrostatic charging and ensure optimal image resolution.



**Figure 2** The preparation process of grafting TPGDA onto WTD.

### 3. RESULT AND DISCUSSION

#### 3.1. Grafting Process of TPGDA onto WTD

The grafting process was conducted by modifying the absorbed radiation dose, TPGDA concentration, reaction time, and reaction temperature. A correlation between GY and the grafting conditions was examined to determine the optimal conditions for WTD grafting. It was anticipated that, under the reaction conditions employed in this study, the relative amount of TPGDA attached onto the surface of WTD would vary accordingly.

GY was investigated in this study with different absorbed radiation doses, as illustrated in Figure 3. The results indicate an increase in GY with an increasing absorbed radiation dose, attributed to the creation of more free radicals. Subsequently, TPGDA monomers diffuse toward the surface of the WTD rubber backbone to interact with the free radicals. This trend aligns with observations from other grafting reactions documented by previous researchers [24-26], wherein a higher absorbed radiation dose provides a greater concentration of radicals and active sites within the polymeric matrix, thereby enhancing radical utilization in graft initiation and chain propagation, leading to a higher grafting yield.

However, a decreasing trend was observed beyond 60 kGy, reaching nearly a plateau beyond 80 kGy. These observations are consistent with findings reported by Xu et al. (2018) [13]. This decline is attributed to termination reactions occurring at an increasing rate relative to their dynamic concentration, ultimately resulting in the leveling off of reactive rubber within the WTD. Furthermore, the plateauing of GY at high absorbed radiation doses may be attributed to free radicals reaching a constant concentration, wherein no further active sites can be formed for polymerization. The grafting extensions tend to

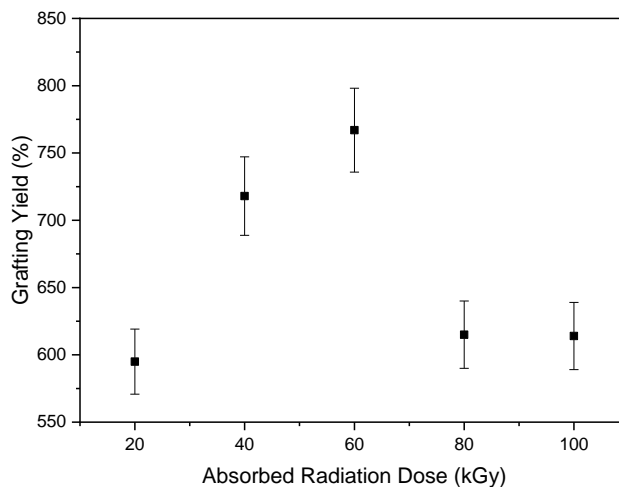
stabilize at higher absorbed radiation doses, likely due to the reaction transitioning into a diffusion-controlled process and experiencing radical fatigue [27].

The relationship between different monomer concentrations was plotted against GY, as shown in Figure 4. Monomer concentration plays a crucial role in the grafting process, as during the radiation-induced grafting reaction, the monomer polymerizes with the free radicals present in the system.

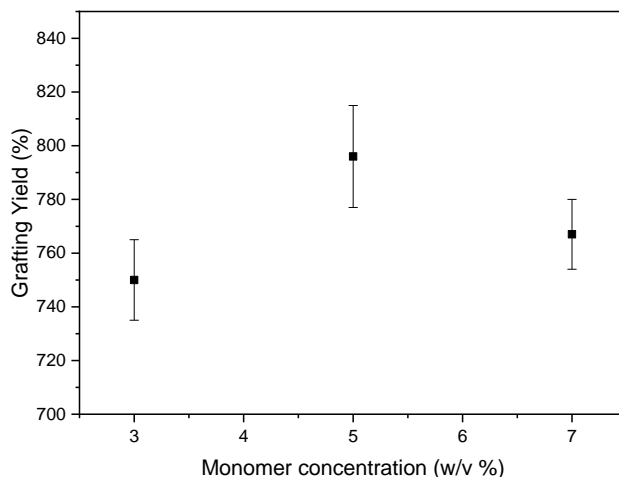
Figure 4 illustrates that the GY initially increases to an optimum value with the rise in TPGDA concentration from 3 w/v% to 5 w/v%. This behavior can be elucidated by the increasing number of TPGDA molecules with the concentration of TPGDA, leading to more available TPGDA molecules in the grafting medium to diffuse to the free radical sites on the WTD rubber backbone. Consequently, there is an abundance of monomer for grafting [28-29]. Particularly at 5 w/v% concentration, a higher availability of TPGDA monomer molecules consumes a significant portion of the free radicals generated during initiation and propagation processes around the WTD rubber, resulting in the highest GY value for this grafting parameter. Thakur et al. (2013) also noted that increasing monomer concentration enhances grafting yield, attributed to more radicals reaching the polymer backbone and forming longer chains, thereby increasing the grafting percentage [28].

However, at higher TPGDA concentrations, the GY levels off due to increased viscosity of the bulk grafting mixture. Elevated viscosity restricts monomer migration to the grafted chain, leading to a decrease in GY. Moreover, the viscosity increase is caused by an excess of monomer in the grafting medium, resulting in increased homopolymer formation, as evidenced by the elevated viscosity of the grafting residues. These findings are consistent with prior research [13], [30-31], wherein the degree of grafting

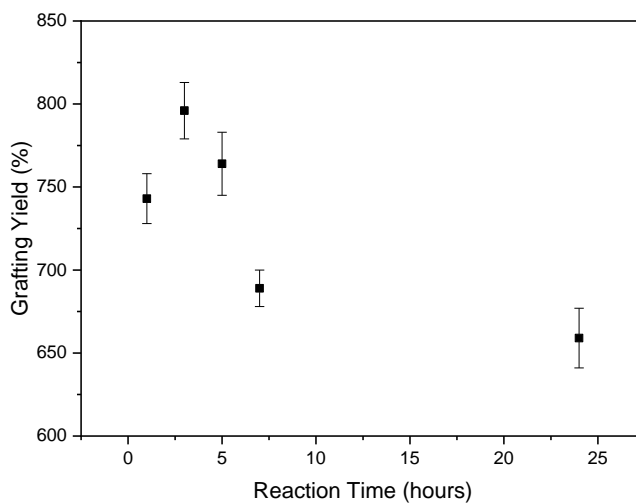
increases with rising monomer concentration and gradually decreases after reaching the maximum monomer concentration that can effectively participate in the grafting reaction.



**Figure 3.** Effect of absorbed radiation dose on GY.



**Figure 4.** Effect of different TPGDA concentration on GY.



**Figure 5.** Effect of reaction time on GY.

A study on the effect of reaction time on GY for grafting TPGDA onto WTD rubber was conducted, and the relationship between them is depicted in Figure 5. The results demonstrated an increase in GY with prolonged reaction time during the grafting process. GY gradually increased from 1 hour to 3 hours of reaction time. This trend can be attributed to the initial enhancement of TPGDA diffusion to the grafting sites, leading to increased availability and involvement of more monomer molecules in the grafting medium [32]. This phenomenon suggests that at 3 hours of reaction time, the highest number of free radicals is created on the WTD rubber backbone, along with the highest availability of TPGDA molecules. Consequently, the grafting reaction proceeds optimally within the system at this given time, resulting in the highest GY value.

Following the optimum reaction time of 3 hours, the decrease in GY may be attributed to the depletion of radicals, resulting in a slower grafting reaction and reduced incorporation of TPGDA molecules onto the WTD rubber [26]. Additionally, as the grafting reaction progresses, mutual annihilation of growing polymeric chains occurs, leading to decreased monomer diffusion and lower GY values. Similar trends have been observed by other researchers regarding the effect of reaction time on GY, where the degree of grafting (DOG) increases rapidly with increasing reaction time until reaching optimum conditions, after which it begins to level off and eventually reaches saturation [31].

The influence of various reaction temperatures on the GY of TPGDA onto WTD rubber is illustrated in Figure 6. GY increased with rising temperature up to 60 °C. A maximum GY of 930 % was observed at 60 °C when comparing GY obtained at 40 °C to 60 °C reaction temperatures. However, upon further increase in temperature from 70 °C to 80 °C, a slight decrease in GY was observed, approximately 22.4 % lower than the maximum GY at 60 °C. This increasing trend can be attributed to higher radical reactivity and molecular motion at elevated temperatures, resulting in increased chain mobility and enhanced monomer diffusion to the grafting region [33]. Consequently, initiation and propagation rates increased, delaying the termination reaction and providing more monomers for reaction with free radicals on the WTD backbone, thereby increasing GY.

Beyond 60 °C, GY decreased with further temperature increase. This decreasing trend at higher temperatures can be attributed to mutual recombination of trapped radicals on the WTD rubber backbone before reacting with diffused monomer molecules. Additionally, bimolecular termination of the graft-growing chains may be enhanced at higher temperatures [34]. Since high temperatures can accelerate radical recombination or transformation, heating can also be utilized to terminate radicals created during irradiation. Consequently, the concentration of radicals decreases as temperature increases. This explains the declining amount of grafted monomer onto the polymer backbone at higher reaction temperatures due to a reduction in the number of free radicals and a slower grafting reaction, resulting in decreased incorporation of TPGDA onto the WTD rubber.

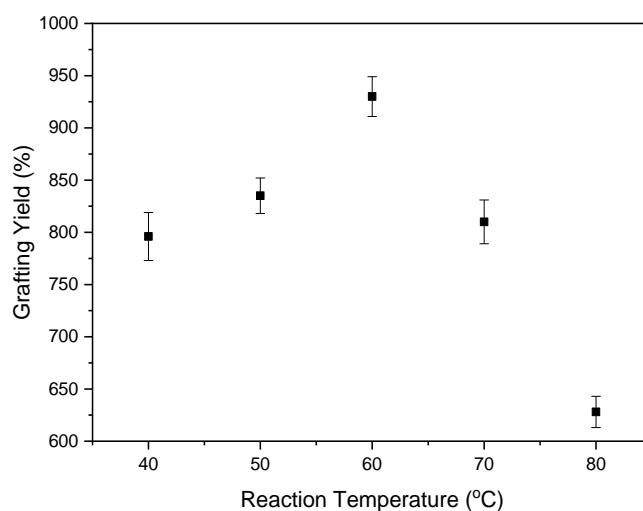


Figure 6. Effect of reaction temperature on GY.

### 3.6. Evidence of TPGDA Grafted WTD Powder

Verification of the presence of the monomer, tripropylene glycol diacrylate (TPGDA), grafted onto the trunk polymer, WTD, is essential. FTIR spectroscopy was employed to analyze the infrared absorption spectra of the functional groups for TPGDA, non-grafted WTD, and grafted WTD, thereby investigating the evolution of WTD surface modification through radiation-induced grafting

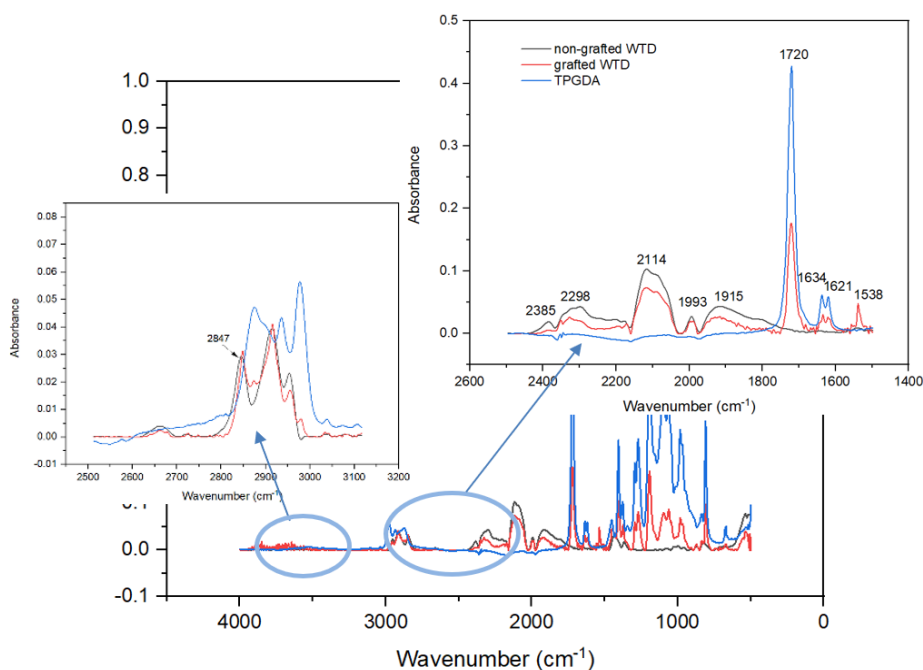
techniques. The spectra are compared and presented in Figure 7.

The FTIR analysis of TPGDA reveals a prominent characteristic peak at 1720  $\text{cm}^{-1}$ , assigned to the carbonyl group ( $\text{C}=\text{O}$ ) originating from the acrylate group. Additionally, peaks at 1099  $\text{cm}^{-1}$  and 1091  $\text{cm}^{-1}$  correspond to the C-O stretch peak, respectively. The presence of C-O-C is indicated by stretching bands at 1271  $\text{cm}^{-1}$  and 1193  $\text{cm}^{-1}$ . The  $-\text{CH}_3$  group contributes to an absorbance peak

observed at  $2977\text{ cm}^{-1}$  in the TPGDA spectrum. These FTIR peaks are consistent with those reported in previous studies utilizing TPGDA [35–37]. For instance, He *et al.* (2017) observed characteristic peaks of TPGDA at  $1720\text{ cm}^{-1}$  and around  $1100\text{ cm}^{-1}$ , corresponding to the C=O and C-O groups, respectively.

Non-grafted WTD rubber primarily comprises hydrocarbons, resulting in predominant methyl and methylene bonds observed in the spectra. The characteristic peaks of non-grafted WTD reveal two groups

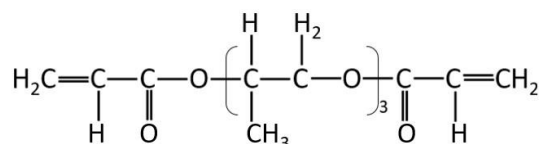
of sharp peaks: (1)  $2114\text{ cm}^{-1}$ ,  $2298\text{ cm}^{-1}$ , and  $2385\text{ cm}^{-1}$ , corresponding to the carbon-carbon double bond (C=C), and (2)  $2953\text{ cm}^{-1}$ ,  $2913\text{ cm}^{-1}$ , and  $2847\text{ cm}^{-1}$ , associated with the stretching vibration of the methylene group (-CH<sub>2</sub>-) present in styrene-butadiene-styrene (SBS), styrene-butadiene rubber (SBR), and isoprene. Peaks at  $1457\text{ cm}^{-1}$  and  $1915\text{ cm}^{-1}$  represent smaller methyl and aromatic compound molecules. This finding aligns with previous reports by Silva *et al.* (2020) and Martínez-Barrera *et al.* (2020), demonstrating similar vibration patterns of waste tire rubber [38–39].



**Figure 7.** FTIR spectra of TPGDA, non-grafted WTD and grafted WTD.

Meanwhile, the FTIR spectra of grafted WTD exhibit similarities with raw WTD, along with several new additional peaks resulting from the grafting process. Notably, several major absorption peaks resemble those of the TPGDA monomer. In this study, the TPGDA monomer

was introduced to WTD rubber in an inert environment. Consequently, the insertion of TPGDA functional groups onto the surface of the WTD backbone is anticipated. The chemical structure of the TPGDA monomer utilized in the grafting process is depicted in Figure 8.



**Figure 8.** Chemical structure of TPGDA.

The comparison of the FTIR spectra of non-grafted and grafted WTD reveals significant differences, providing evidence for the successful grafting of TPGDA onto the WTD rubber backbone. In the grafted WTD spectra, peaks corresponding to the =C-H deformation of vinylidene hydrocarbon and the >C=C< stretching for the vinylidene group are observed at  $983\text{ cm}^{-1}$  and  $808\text{ cm}^{-1}$ , respectively. Additionally, a new peak appears at  $1096\text{ cm}^{-1}$  corresponding to the C-O stretch peak, and stretching bands at  $1193\text{ cm}^{-1}$  and  $1272\text{ cm}^{-1}$  indicate the presence of C-O-C bonds. The absorption bands at  $1621\text{ cm}^{-1}$  and  $1634\text{ cm}^{-1}$

are related to the C=C group. Notably, a strong peak at  $1720\text{ cm}^{-1}$  attributed to the carbonyl vibration from TPGDA is evident in the grafted WTD spectra but absent in the non-grafted WTD spectra. Since any copolymer, homopolymer, and unreacted monomer were removed during the reaction, this finding provides sufficient evidence to confirm that TPGDA has been grafted to WTD rubber backbones.

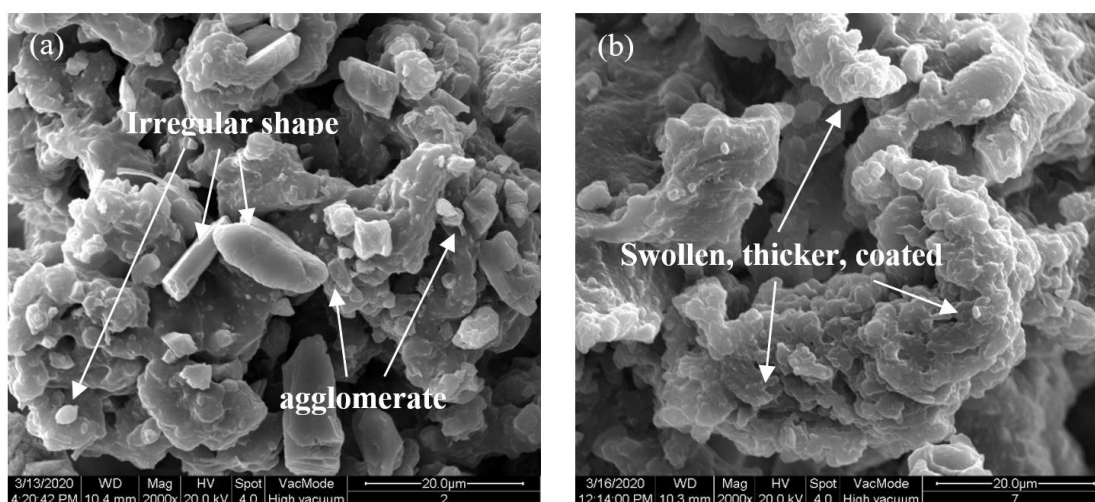
Furthermore, the FTIR spectra of grafted WTD also exhibit peaks at  $2847\text{ cm}^{-1}$ ,  $2915\text{ cm}^{-1}$ , and  $2953\text{ cm}^{-1}$  related to the stretching vibration of the methylene group, with similar



intensity to non-grafted WTD. This suggests that the grafting of the TPGDA monomer does not significantly affect the methylene group in this spectral range. However, the intensity of peaks at  $2114\text{ cm}^{-1}$ ,  $2298\text{ cm}^{-1}$ , and  $2385\text{ cm}^{-1}$  is reduced after the grafting process, indicating interaction between the TPGDA monomer and the WTD rubber backbone facilitated by the free radicals created during the irradiation process. Although no carbonyl group existed in the non-grafted WTD, a carbonyl peak appears in the grafted WTD spectra. The strong absorption peak of this carbonyl characteristic peak in grafted WTD indicates that the TPGDA monomer has indeed reacted with the free radicals on the surface of WTD rubber. Moreover, the presence of several new peaks further supports the FTIR data, providing further evidence for the successful grafting of the TPGDA monomer onto the WTD rubber backbone.

The effect of TPGDA grafting on the surface morphology of WTD rubber was studied using SEM, confirming the

changes observed in the FTIR analysis. The SEM micrograph of non-grafted WTD shows irregular shapes with small particle agglomerates, albeit smaller in size compared to the grafted WTD rubber samples. In contrast, the SEM micrograph of grafted WTD reveals a rougher, thicker, swollen, and coated surface, indicating successful grafting of TPGDA onto the WTD rubber surface. Moreover, through the incorporation of the TPGDA monomer, the average diameter of raw WTD rubber increased by approximately 111.5%, from  $8.7 \pm 2.4\ \mu\text{m}$  to  $18.4 \pm 4.2\ \mu\text{m}$ . This comparison of micrographs clearly demonstrates that the surface morphology of raw WTD rubber changes upon grafting, indicating successful grafting of a considerable amount of TPGDA onto WTD rubber samples under optimal grafting conditions. Therefore, the grafting procedure was indeed successful, as evidenced by the noticeable increase in particle size of the WTD rubber sample.



**Figure 9.** SEM images of (a) non-grafted WTD (b) grafted WTD.

#### 4. CONCLUSION

The successful grafting of emulsion TPGDA onto WTD has indeed been demonstrated, achieving a remarkable GY of 930%. The radiation-induced grafting technique employed in this study proved effective, particularly with the optimized parameters: TPGDA concentration of 5 w/v%, irradiation dose of 60 kGy, reaction time of 3 hours, and reaction temperature of  $60\text{ }^{\circ}\text{C}$ .

Verification of the presence of TPGDA monomer grafted onto the WTD trunk polymer is crucial, and this was accomplished through thorough analysis of the physicochemical properties of the grafted WTD. Chemical identification using FTIR spectroscopy and morphology analysis using SEM were instrumental in confirming the success of the grafting process.

The FTIR spectra provided compelling evidence of TPGDA monomer reacting with the free radicals on the surface of WTD rubber, as indicated by the emergence of a new peak

at  $1720\text{ cm}^{-1}$  attributed to the carbonyl group from the TPGDA monomer. This finding was further corroborated by SEM analysis, which revealed that the surface morphology of grafted WTD had become thicker, swollen, and coated compared to non-grafted WTD. Additionally, the incorporation of TPGDA monomer led to a significant increase in the average diameter of the non-grafted WTD particles, further validating the success of the grafting process.

In summary, the comprehensive analysis conducted in this study confirmed the successful grafting of TPGDA monomer onto WTD rubber samples under the optimized grafting conditions, thereby opening up possibilities for the utilization of grafted WTD in various practical applications.

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