Materials Research. 2025; 28: e20240618
DOI: https://doi.org/10.1590/1980-5373-MR-2024-0618

Effect of Surface Crosslinking on the Wear and Friction Behavior of Tea Polyphenol Stabilized Ultra-High Molecular Weight Polyethylene for Total Joint Replacements

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Received: Dezembro 30, 2024; Revised: Abril 8, 2025; Accepted: Maio 29, 2025

Highly crosslinked ultra-high molecular weight polyethylene (UHMWPE) with vitamin E (VE) as an antioxidant is widely used for joint implants. VE helps resist oxidation but raises concerns about reduced crosslink density and wear performance. This research hypothesized that tea polyphenols (tPPs), such as lipid-soluble epigallocatechin gallate (lsEGCG) and epigallocatechin gallate (EGCG), could prevent the usual decrease in crosslink density caused by VE, thereby enhancing wear performance. The antioxidants were blended with UHMWPE at 0.2 wt% and surface chemically crosslinked using di-cumyl peroxide. The surface properties, including crosslink density, roughness, coefficient of friction, and wear performance, were evaluated in detail. The results showed that lsEGCG and EGCG blended UHMWPE had significantly higher crosslink density compared to VE stabilized UHMWPE, which was 17% lower than virgin UHMWPE. The coefficient of friction increased after crosslinking and was higher in tPPs blended UHMWPE, indicating a highly crosslinked network structure. The wear resistance of surface crosslinked tPPs stabilized UHMWPE was significantly higher than VE-stabilized UHMWPE. Additionally, a substantial number of scratches, furrows, and flakes were observed on the surface of VE-stabilized UHMWPE compared to tPPs stabilized UHMWPE. It was concluded that tPPs are promising alternatives to VE for improving the performance and longevity of UHMWPE-based implants.

Keywords: *Highly cross-linked UHMWPE, Vitamin E and Tea polyphenols, Wear performance, Surface crosslinking, Total joint replacement.*

1. Introduction

The rapid increase in joint patients has made it necessary for researchers to develop high-performance biomaterials used in total joint replacement (TJR)[1]. TJR is a rapidly growing market, driven by factors such as an aging population, increasing prevalence of arthritis and joint injuries, and advancements in technology and materials used in joint replacement surgeries. According to a market research report by Grand View Research[2], the global TJR market was valued at USD 22,474.6 million in 2024 and is expected to grow at a compound annual growth rate (CAGR) of 4% to USD 28,698.0 million by 2030. Meanwhile in comparison, the Brazil joint replacement market generated a revenue of USD 242.8 million in 2022 and is expected to reach USD 353.7 million by 2030. The Brazil market is expected to grow at a CAGR of 4.8% from 2023 to 2030[3].

Ultra-high molecular weight polyethylene (UHMWPE) with further modification, has been a material of choice for total hip and total knee replacement with excellent results for the last two decades[4,5]. A high-performance implant has three major factors: wear, mechanical strength, and oxidation stability. The dominant cause of joint failure is wear and tear in the polymer component due to constant movement and contact parts leading to joint loosening and osteolysis[6-8]. Crosslinking has been widely accepted to enhance wear resistance, by creating a network structure to minimize the surface orientation and alter the surface topography, playing a role in releasing wear debris in UHMWPE[9,10]. Radiation crosslinking has displayed promising results in decreasing the wear of UHMWPE in artificial joints, due to highly crosslinked UHMWPE structure achieved through radiation crosslinking by using high-energy ionizing gamma radiation or electron beam[11]. However, radiation crosslinking causes oxidation embrittlement due to the formation of free

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radicals reacting with oxygen, leading to degradation in the mechanical performance of the implant[12,13]. Crosslinking by radiation produces free radicals by splitting the UHMWPE bonds. The free radicals formed in the amorphous phase are turned into a crosslinked structure while the free radicals created in the crystalline region highly affect the long-term oxidation resistance capabilities of the polymer[14].

Chemical crosslinking has been in use for several decades to achieve a highly crosslinked structure using organic peroxides, but its oxidizing nature hindered its use in clinical applications[15]. Antioxidants are widely used to enhance the oxidation resistance of the crosslinked UHMWPE[16,17]. Vitamin E (VE) is highly preferred as a natural antioxidant, providing a significant enhancement in oxidation resistance[18]. VE donates hydrogen from the phenolic group to react with the trapped residual radicals playing a role in hindering chain scission[19]. However, VE is also a free radical scavenger and it decreases the crosslinked density of the polymer, resulting in reducing the effectiveness of wear resistance of the crosslinked UHMWPE[20,21]. To reduce the inhibitive effect on the crosslinked structure, ~ 0.2 wt% of VE content is considered an optimal amount evaluated by Oral et al.[15]. Therefore, the current research is focused to investigate an effectual stabilizer along with the minimum adverse effect on the crosslinked structure.

Recent research has explored a more powerful antioxidant, tea polyphenol found in tea as a natural substance, which showed a stronger resistance to oxidation than VE and is regarded as a better alternative stabilizer[22-24]. Tea polyphenols have the capacity for higher hydrogen donation due to their many phenolic hydroxyls compared to VE, which is comprised of single phenolic hydroxyl. In addition to that, tea polyphenols incorporation does not affect the crosslinking process compared to the decrease occurring in VE blended UHMWPE[25]. Further, the connections between friction, wear, and crystallinity are still not fully understood. Gaining insights into the influence of structure and crystallinity on friction and wear properties would be beneficial for the advancement of high-quality joint materials[26,27].

This investigation posits that the utilization of tea polyphenols as antioxidants, specifically lipid-soluble epigallocatechin gallate (lsEGCG) and epigallocatechin gallate (EGCG), instead of vitamin E, can augment the wear resistance of ultra-high molecular weight polyethylene (UHMWPE). This substitution is anticipated to maintain an elevated crosslink density and result in heightened oxidation stability. The chosen polyphenols, lsEGCG and EGCG, were selected based on their commendable microbial inhibition and oxidation stability characteristics[23,28]. Subsequent to independent blending with UHMWPE, the blends underwent surface crosslinking employing di-cumyl peroxide (DCP). The study encompassed an assessment of crosslink density, coefficient of friction, and wear rate of the crosslinked antioxidant-blended UHMWPE. Furthermore, an analysis of the crystalline structure and surface topography was conducted.

2. Materials and Methods

2.1. Materials

UHMWPE (GUR1050; Ticona, UK) with an average molecular weight of ~5×10^6 g/mol, were used in this study. VE was obtained from Aladdin, China while the tea polyphenols (lsEGCG and EGCG) were procured from Nanjing Zhu Biological Technology Co. Ltd. Di-cumyl peroxide (DCP) was procured from Alfa Aesar, Germany. The chemical structures for all the chemicals are given in Table 1.

2.2. Preparation of UHMWPE blends

Antioxidants were separately added into 100 gm UHMWPE at a fraction of 0.2 wt% in a round bottom flask containing 120 ml isopropyl alcohol. The materials were constantly stirred at 200 rpm using a mechanical stirrer at room temperature for 24 hrs. The blends were then dried in a vacuum oven for 7 days at a temperature of 60 °C under a vacuum environment. It was followed by consolidation of the blends in compression molding at 200 °C and 10 MPa. The compression cycles of 3 min were repeated for a total of 20 min at the same temperature and pressure. It is then cooled at room temperature for 10 min under the same pressure (10 MPa). Three Pucks of 108 mm in diameter and 10 mm in thickness were produced for each category of blends. The pucks were further machined to a smaller disc of 40 mm diameter and 4 mm thickness which is the required dimension for the pin-on-disc tribometer. Virgin UHMWPE (UH), VE blended (VE-UH), lsEGCG blended (lsEGCG-UH) and EGCG blended UHMWPE (EGCG-UH) were the four different categories of samples prepared for the comparative analysis. For simplicity, the UHMWPE is shortened to UH.

2.3. Chemical crosslinking using peroxide

DCP is an organic peroxide with a melting temperature of 38 °C is a solid and used for achieving the surface crosslink structure. The initial step was the diffusion of the peroxide by keeping the samples in the peroxide in a 200 ml round bottom flask in an oil bath. The doping temperatures were kept at 80 °C and a constant nitrogen flow was ensured throughout the process for 4 hrs. Decomposition of the peroxides in the samples is a subsequent process by heating the samples at 150 °C in an empty flask for 4 hrs in a nitrogen environment. The experimental parameters are the best possible values previously reported by Gul et al.[28]. The surface crosslinking schematic diagram is depicted in Figure 1a.

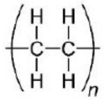
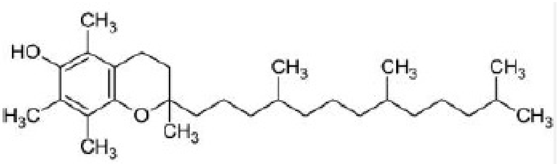
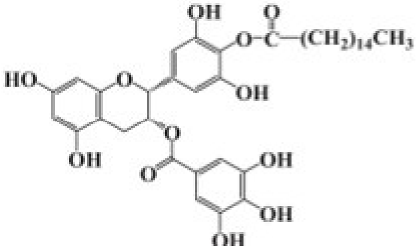
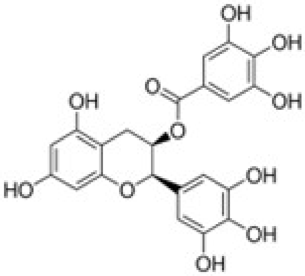
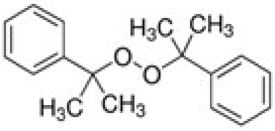
2.4. Percent weight change before and after crosslinking

The weight of the samples was measured before and after the diffusion process, and at the end of the decomposition process using a weighing scale (Shimadzu Corporation ATX224). The weight change after the diffusion step was considered as the peroxide intake. Weight changes after the decomposition process imply the decomposition of the peroxide and the crosslinked structure developed in the polymer.

2.5. Crosslink density measurement by gravimetric swelling method

To measure the surface crosslink density of different UHMWPE blends, small cubes were carefully cut from the exterior part of the crosslinked samples. Each cube had a thickness of 1 mm with a cross-sectional dimension of 3 × 3 mm. For every group, four samples (n = 4) were

Table 1. Molecular formula and chemical structures of the chemicals used in this work.

Chemical	Abbreviation	Molecular formula	Chemical structure
Ultra-high molecular weight polyethylene	UHMWPE	$(C_2H_4)n$	
Vitamin E (alpha tocopherol)	VE	$C_{29}H_{50}O_2$	
Lipid soluble epigallocatechin gallate	IsEGCG	$C_{38}H_{48}O_{12}$	
epigallocatechin gallate	EGCG	$C_{22}H_{18}O_{11}$	
Di-cumyl peroxide	DCP	$C_{18}H_{22}O_2$	

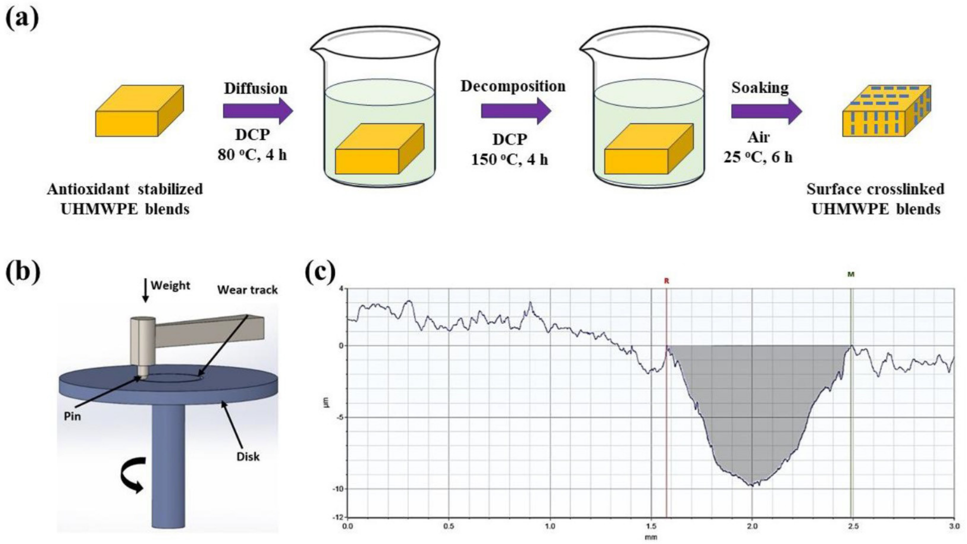


Figure 1. (a) Schematic diagram of the surface crosslinking of UHMWPE blends using DCP. (b) Friction and wear testing setup by pin-on-disc tribometer. (c) Cross section area of the wear track was measured by a profilometer.

prepared for analysis. These sections were then subjected to a swelling test to determine the crosslink density at the surface. The cubes swelled in xylene (DAEJUNG chemicals & metal, China) at 130 °C and kept for 2 hrs to attain equilibrium status. The gravimetric swell ratio was calculated from its initial weight and the xylene uptake, which were used to measure the crosslink density by using a previously reported process as shown in Equation 1[29].

$$d_x = -\frac{\ln\left(1-q_{eq}{}^{-1}\right)+q_{eq}{}^{-1}+Xq_{eq}{}^{-2}}{V_1 q_{eq}{}^{-\frac{1}{3}}} \tag{1}$$

where,

$$q_{eq} = \left(\frac{h_f}{h_i}\right)^3$$

$$X = 0.33 + \frac{0.55}{q_{eq}}$$

and,

$$V_1 = 136\ cm^3 / mol$$

In the above equations, q_{eq} which is the apparent volume swelling ratio, was calculated using the height of the test cube before (h_i) and after (h_f) the equilibrium swelling is reached. In addition, V_l is the molar volume of the solvent (xylene) at 130 ºC and X is the polymer solvent interaction parameter at 130 ºC.

2.6. Melting point and crystallinity analysis using differential scanning calorimetry

Differential scanning calorimeters (DSC 800, Perkin Elmer, USA) were employed to generate the first melting profile of un-crosslinked UHMWPE and its blends. Samples (~5 mg) were heated from 40 to 180 °C at a heating rate of 10 °C/min while ensuring the nitrogen gas atmosphere. The percent crystallinity is measured by integrating the enthalpy peak from 40 to 160 °C and normalizing it with the enthalpy of fusion of 100% crystalline polyethylene (289.3 J/g, as per ASTM F2625-10 (2016) standard), as shown in Equation 2.

$$X_c = \frac{\Delta H_f}{\Delta H_f^o} \times 100\ \% \tag{2}$$

Where, X_c is the degree of crystallinity (%), ΔH_f is the measured heat of fusion of the sample (J/g) from the DSC curve and, ΔH_f^0 is the theoretical heat of fusion for 100% crystalline polyethylene[30].

2.7. Measurement of co-efficient of friction by pin-on-disc (POD) tribometer

Universal and Versatile Pin-On-Disc & Oscillating Tribometer (POD; Tt-Tribotechnic, Clichy, France) was used to study the friction and wear behavior of the polymer blends. Figure 1b illustrates the schematic of friction and wear testing methodology adopted to test UHMWPE samples. The pin was consisting of an aluminum oxide ball (2 mm diameter), which was tested against the disc's samples (4 mm thickness and 40 mm diameter, n = 4) in diluted bovine serum (25%, SAFC Sigma-Aldrich, Buch, Switzerland) as a natural lubricant for each category of blends. A 10 N static load were exerted by the pin on the moving discs (60 mm/sec, 3448 cycles) at a 2 mm radius from its center for 3 hrs. This load is equivalent of 3.18 MPa of contact pressure replicating the minimum pressure exerted on UHMWPE load bearings in total joint replacements[31]. This process was repeated for all the samples.

2.8. Determination of wear rate by mechanical profilometer

The wear rate of virgin UHMWPE, VE-UH, IsEGCG-UH and EGCG-UH samples was calculated from the wear track created by the pin-on-disc tribometer, using a mechanical profilometer (DektakXT stylus Profilometer, Bruker Company, Massachusetts, U.S). The samples were attached to the profilometer table and the stylus was positioned near the wear track, seen through the device camera by a visual user interface. The wear track curve was generated through a contact stylus to calculate the cross-sectional area (Figure 1c). Finally, the wear rate was calculated from the wear track curve. The tests were repeated three times.

2.9. Surface characterization using scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) analysis was performed using a Zeiss EVO® LS 10 microscope (Oberkochen, Germany). The electron beam energy was set at 15 kV, and imaging was conducted in secondary electron (SE) mode. Prior to scanning, all samples were prepared using an ultra-microtome to obtain smooth surfaces for analysis. To enhance conductivity, a thin film of gold (Au) was sputter-coated onto the samples. The samples analyzed included crosslinked and uncross linked UHMWPE for the following four categories: Virgin UHMWPE (UH), VE-blended UHMWPE (VE-UH), lsEGCG-blended UHMWPE (lsEGCG-UH), EGCG-blended UHMWPE (EGCG-UH).

2.10. Surface roughness measurement

The samples were cleaned in an ultrasonic cleaner and carried out for surface roughness measurement through an Accretech mechanical profilometer (ACCRETECH SBS, UK Ltd). The profilometer having a stylus of 4 mm was positioned in the center to move over the sample's surfaces at a speed of 0.5 mm/sec by taking 400 mm of evaluation length. Three scans were taken for each of the samples by changing the position after every scan. A total of 32 samples were analyzed using the profilometer, with four samples per category across eight material categories. These included both crosslinked and un-crosslinked variations of: Virgin UHMWPE (UH), VE-blended UHMWPE (VE-UH), lsEGCG-blended UHMWPE (lsEGCG-UH), EGCG-blended UHMWPE (EGCG-UH). All sample categories were examined to ensure a comprehensive evaluation of surface characteristics.

2.11. Statistical analysis

Student's *t*-test was performed as a statistical analysis method for the two-tailed distribution of unequal variance and assigning the significance to $p < 0.05$ (n >3).

3. Results

3.1. Weight change of UHMWPE blends

The percent weight gains due to DCP diffusion into the virgin UHMWPE, VE-UH, lsEGCG-UH and EGCG-UH is shown in Figure 2a. The percent weight change for virgin-UH, VE-UH, lsEGCG-UH and EGCG-UH is increased by 1.7 ± 0.02, 1.6 ± 0.04, 1.4 ± 0.04, and 1.5 ± 0.03 respectively after the diffusion. The percent weight change is seen to be higher for virgin-UH compared to other samples after the diffusion process. That describe the weight gain is highest in virgin-UH. The diffusion process is followed by decomposition process at a temperature of 150°C for a time duration of 4h. It was observed that the percent weight change for virgin-UH, VE-UH, lsEGCG-UH, and EGCG-UH is increased by 0.6 ± 0.02, 0.8 ± 0.03, 0.7 ± 0.02, and 0.9 ± 0.02 respectively after the decomposition process. The percent weight change is observed to be higher for EGCG-UH compared to other samples after the decomposition process. This could be attributed to the formation of higher crosslinked network structure in EGCG-UH. The percentage weight loss after the decomposition is highest in virgin-UH (63.9%) while the lowest is observed in EGCG-UH samples, which is 40.1% as shown in Figure 2b.

3.2. Crosslinking density of UHMWPE blends

The crosslinked density of virgin UHMWPE and its blends with 0.2 wt% antioxidants are shown in Figure 2c. The crosslinked density of virgin UHMWPE is 305.4 mol/m^3 (as shown by the red dashed line in Figure 2c). Interestingly, EGCG-UH shows a crosslink density of 309.7 mol/m^3, which is slightly higher than that of virgin UHMWPE. On the other hand, the addition of VE into the UHMWPE blends causes a massive reduction in the crosslinked density. The crosslinked density of VE-UH is 254 mol/m^3 with a noticeable decrease of 17%. In comparison to virgin UHMWPE, the crosslinked density of tea polyphenols blended samples remained unchanged. The crosslinked density of EGCG-UH and lsEGCG-UH is 295 mol/m^3 and 309 mol/m^3 respectively that show a considerable difference compared with the VE-UH, rather higher than virgin UHMWPE ($p < 0.05$). It has been shown previously that crosslinking creates a cage mechanism and meeting points contain peroxide molecule[32]. The crosslinked structure is formed at the entanglement site due to the compactness between polymer chains. The crosslinked density increase observed in EGCG-UH and lsEGCG-UH is a result of higher entanglement density in tea polyphenols due to its potential for the removal of free radicals.

3.3. Surface Roughness

Un-crosslinked and virgin UHMWPE shows the lowest surface roughens value of 0.626 µm compared with tea polyphenols blended samples, which have the highest surface roughness values of 1.094 µm and 1.071 µm for lsEGCG and EGCG blended UHMWPE respectively (Figure 2d). The surface roughness value of VE-UH is 0.76 µm. However, no significant difference in the results of the crosslinked and un-crosslinked VE-UH samples was observed, which was comparable to the crosslinked virgin UHMWPE ($p < 0.05$).

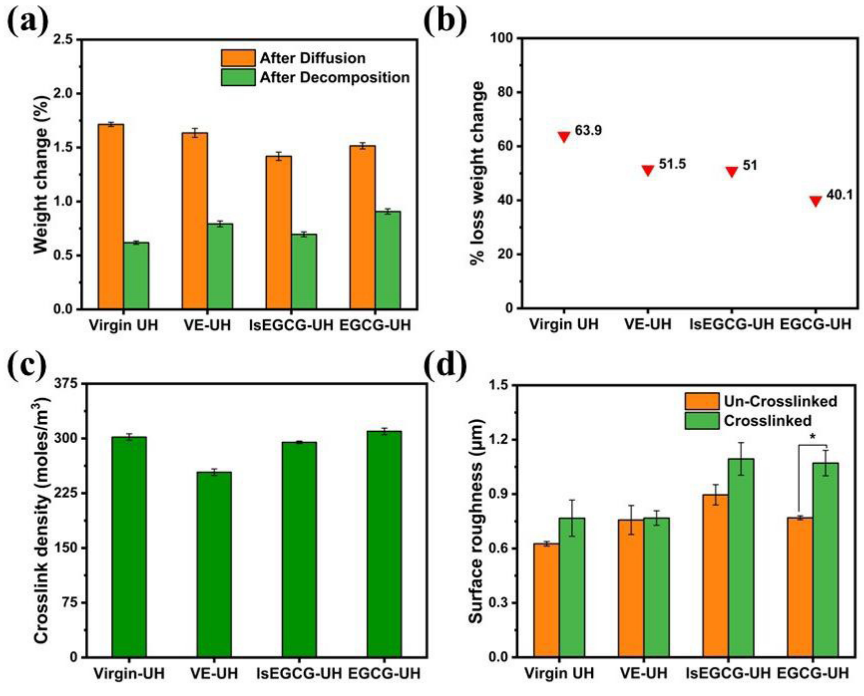


Figure 2. (a) Percent weight gain after doping at 80 °C with DCP and decomposition at 150 °C for virgin, VE, lsEGCG and EGCG blended UHMWPE. (b) Comparison of percent difference in weight change between different UHMWPE blends after the decomposition process. (c) Crosslink density of VE, lsEGCG and EGCG blended UHMWPE, the red dashed line shows the crosslink density of virgin UHMWPE blends. (d) Surface Roughness of different crosslinked and un-crosslinked UHMWPE blends. (*$p < 0.05$).

3.4. Melting point and crystallinity of UHMWPE blends

Melting point (T_m) and crystallinity (X_c) of the crosslinked and antioxidants blended samples were evaluated by the first heating profiles as shown in Figure 3a. It is observed that the melting curve widens for virgin UHMWPE and lsEGCG-UH, while it is seen sharper in VE-UH and EGCG/UHMWPE. X_c of virgin UHMWPE, 0.2 wt% VE blended, 0.2 wt% lsEGCG blended and 0.2 wt% EGCG blended UHMWPE are shown in Figure 3b. The X_c of crosslinked UHMWPE is 32% in the absence of antioxidants, which is seen to be higher among all the other samples. In contrast, X_c of lsEGCG blended UHMWPE is 27%, which is significantly lower compared to VE-UH and EGCG-UH ($p < 0.05$). The T_m of VE-UH and lsEGCG-UH were found to be nearly identical, at 149 °C and 148 °C respectively, as shown by the highest points on the line in Figure 3c. There was no significant difference between the T_m for each blend ($p > 0.05$)

3.5. Friction behavior of UHMWPE blends

The coefficient of friction (COF) values and wear rate were evaluated to characterize the tribological behavior of virgin UHMWPE, VE-UH, lsEGCG-UH and EGCG-UH samples. Figures 4a and b displays the friction coefficient change of the un-crosslinked and crosslinked group of samples respectively. The friction coefficient of uncross linked VE blended UHMWPE is 0.071, taken as the lowest reference among the whole samples. The friction coefficient of virgin UHMWPE, lsEGCG blended and EGCG blended are 0.081, 0.101 and 0.103 respectively before crosslinking comparing with crosslinked samples of friction coefficient 0.109, 0.074, 0.1201 and 0.172 for virgin UHMWPE, VE-UH, lsEGCG-UH and EGCG-UH ($p < 0.05$). The highest values are observed for the tea polyphenols blended samples; especially EGCG-UH friction coefficient is more than double compared with VE-UH.

3.6. Wear of UHMWPE blends

Figure 5 displays the wear volume of crosslinked and uncross linked specimens. The wear volume of un-crosslinked virgin UHMWPE, VE-UH, lsEGCG-UH and EGCG-UH have minute differences and vary around 0.123-0.13 mm^3. The wear volume decreases significantly after the crosslinking and the result concentrates at 0.086-0.092 mm^3 ($p < 0.05$). The cross-link density achieved on the surface of peroxide

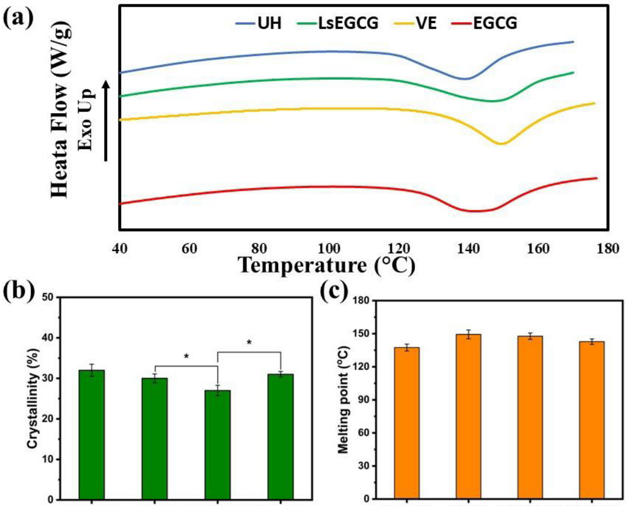


Figure 3. (a) DSC melting profiles of virgin, VE, lsEGCG and EGCG blended UHMWPE, (b) Crystallinity of virgin, VE, lsEGCG and EGCG blended UHMWPE. (* $p < 0.05$) (c) Melting point of virgin, VE, lsEGCG and EGCG blended UHMWPE.

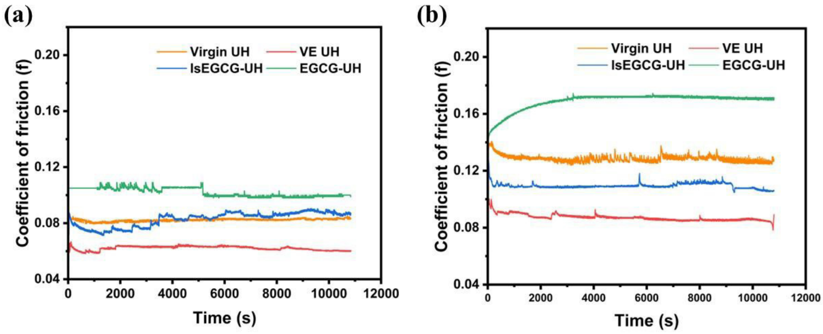


Figure 4. Coefficient of friction for virgin, VE blended, lsEGCG blended and EGCG blended UHMWPE, (a) un-crosslinked, and (b) surface crosslinked.

diffused samples after decomposition resulted in a wear rate of approximately 0.0864 mm^3, 0.0919 mm^3, 0.0856 mm^3 and 0.0865 mm^3 for virgin-UH, VE-UH, lsEGCG-UH and EGCG UH blends, respectively. The UHMWPE blended with lsEGCG exhibited the greatest reduction in wear, measuring 32.6%, while the blend with VE showed the lowest reduction in wear, measuring 29.4% (Figure 5). In comparison, EGCG-UH blends show a decrease in wear rate by 29.5% from 0.1226 to 0.0865 mm^3.

3.7. Scanning electron microscopy

Figure 6 shows the SEM images of the wear track on virgin UHMWPE samples and antioxidants blended UHMWPE samples before and after surface crosslinking. Deep and wide grooves are observed on the surface of uncross linked virgin UH. In comparison, there is significant damage to the

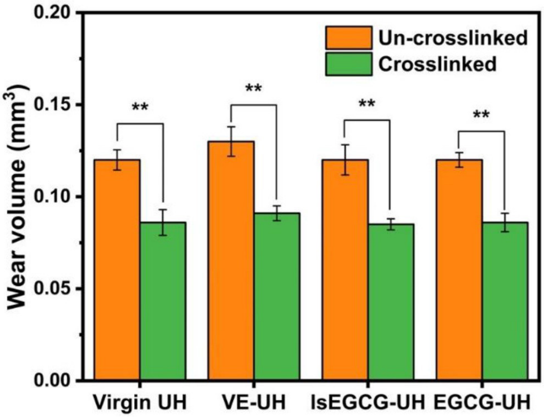


Figure 5. Wear volume of virgin UHMWPE, VE-UH, lsEGCG-UH and EGCG-UH before and after the crosslinking process. (** $p < 0.05$).

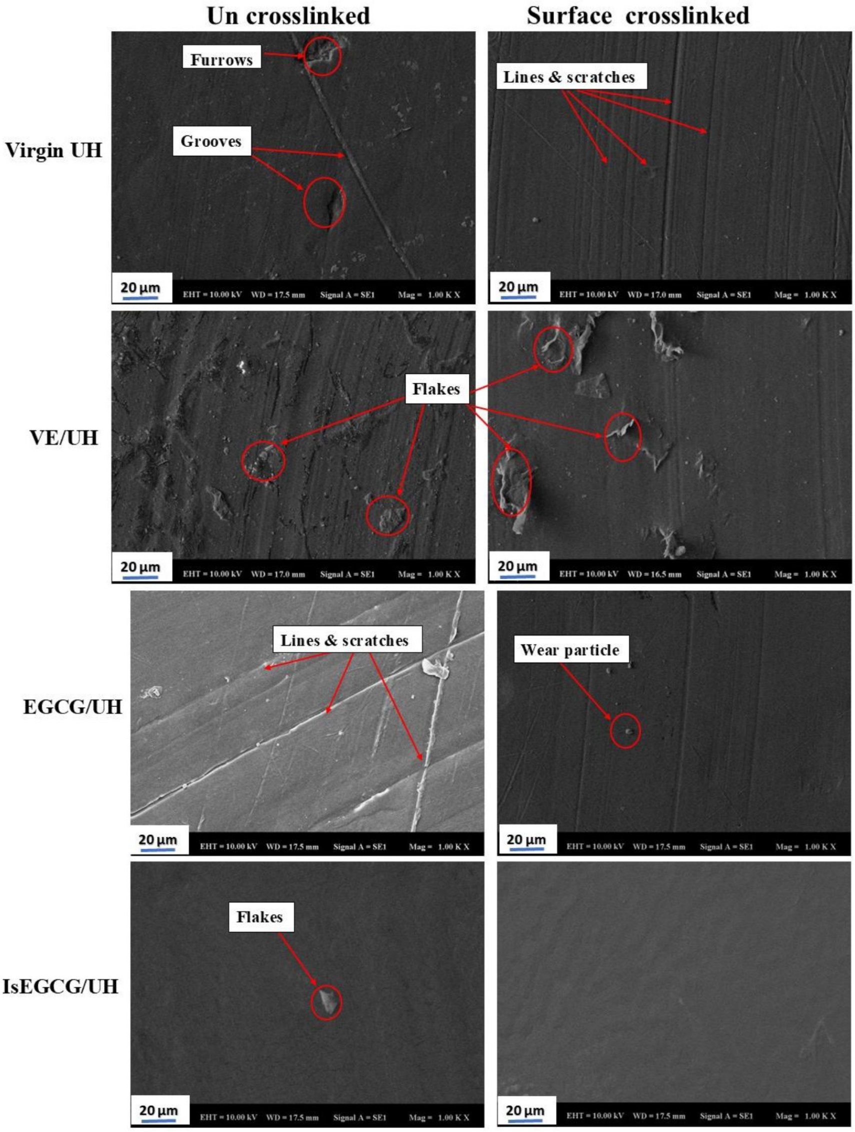


Figure 6. Micrograph of the wear track of different UHMWPE blends before and after surface crosslinking. The red arrows represent furrows, grooves, lines, scratches, flakes and wear particle. The magnification of the images is 1000 x at 20 µm.

surface of VE-UH in the form of delamination and flakes. This could be attributed to the oxidation wear in VE-UH blends due to the reaction of peroxide free radicals. Similar to virgin UH there are few lines and scratches on the surface of EGCG-UH which is significantly reduces after surface crosslinking. A noticeable difference was observed in the lsEGCG-UH blends where the surface appeared smooth and damage free. This indicates that on the surface of the lsEGCG-UH there is a highly crosslinked network structure which is preventing against the abrasion of the UHMWPE against the ceramic ball.

4. Discussion

Chemical crosslinking is generally carried out by blending the peroxides into the resin powder and processing it along the peroxides[33]. Our proposed method of crosslinking used a peroxide with a high enough decomposition temperature in order to allow peroxide diffusion into the polymer. Based on the previous study by Gul et al.[28], we selected DCP as organic peroxide with a 1h half-life temperature of 137 °C[34]. The diffusion temperature was set to 80 °C far below the decomposition of the selected peroxide. Weight gain observed after the decomposition is attributed to the existence of peroxide decomposition products (Figure 2a). Common decomposition products for di-cumyl peroxide are acetone, methane, and cumyl alcohol[35]. The weight gain after doping is a result of peroxide charging into the polymer, while almost 50-65% of the weight gain during diffusion was lost after decomposition due to the volatility of the peroxide (Figure 2a). The peroxide volatile nature after decomposition is essential for the ultimate use of chemical crosslinked UHMWPE as joint implant material[34]. The surface crosslinked structure is accomplished and the results are consistent with the previous research conducted for the same group of samples[23].

Peroxides create free radicals by abstracting hydrogen from the UHMWPE chain during the decomposition process, leading to a crosslinked structure[36]. However, the mechanical strength is undesirably depreciated due to free radicals, which act as an initiator for oxidation embrittlement[14]. VE has been frequently used for oxidation stability and is approved by FDA (Food and Drug Administration) for biomedical purposes. The oxidation mechanism of VE in irradiated UHMWPE involves the donation of hydrogen, which is then scavenged by the proton from VE to prevent the cascade reactions with oxygen and hinder the formation of free radicals[37,38]. The free radicals are highly prone to react with an antioxidant before achieving the crosslinking structure due to the presence of only one phenol hydroxyl resulting in a reduced crosslink density. The previously reported work is verified by the loss of crosslink density with VE blended UHMWPE (Figure 2c). In contrast to VE, the crosslink density of lsEGCG-UH and EGCG-UH blends are increased, approximately equaling the virgin UHMWPE (Figure 4). It is suggested that tea polyphenols contain multiple phenolic groups that can detach many phenoxy radicals from dehydrogenated lsEGCG and EGCG, which participate in crosslinking along with peroxide, therefore, improving the crosslink density[39]. For example, in the case of EGCG, DCP decomposes under heat to form alkoxy radicals that abstract hydrogen (H) atom from UHMWPE.

$(C_6H_5\text{-}C(CH_3)_2\text{-}O)_2 \rightarrow 2\ C_6H_5\text{-}C(CH_3)_2\text{-}O\bullet$

Dicumyl peroxide Alkoxy radicals

$\text{-}[CH_2\text{-}CH_2]_n\text{-} + C_6H_5\text{-}C(CH_3)_2\text{-}O\bullet \rightarrow \text{-}[CH_2\text{-}CH\bullet]_n\text{-} + C_6H_5\text{-}C(CH_3)_2\text{-}OH$

Polyethylene chain Radical site formed

$\text{-}[CH_2\text{-}CH\bullet]_n\text{-} + \bullet CH\text{-}CH_2]\text{-}_n \rightarrow \text{-}[CH_2\text{-}CH\text{-}CH\text{-}CH_2]\text{-}_n$

Crosslinked UHMWPE (C-C bond between chains)

EGCG donates phenolic H to stabilize radicals and form additional crosslinks

$(C_6H_5\text{-}C(CH_3)_2\text{-}O)_2 \rightarrow 2\ C_6H_5\text{-}C(CH_3)_2\text{-}O\bullet$ (Peroxide radicals)

$C_6H_5\text{-}C(CH_3)_2\text{-}O\bullet + \text{-}[CH_2\text{-}CH_2]_n\text{-} \rightarrow \text{-}[CH_2\text{-}CH\bullet]_n\text{-} + C_6H_5\text{-}C(CH_3)_2\text{-}OH$

$\text{-}[CH_2\text{-}CH\bullet]_n\text{-} + EGCG\text{-}OH \rightarrow \text{-}[CH_2\text{-}CH_2]_n\text{-} + EGCG\text{-}O\bullet$

$EGCG\text{-}O\bullet + \text{-}[CH_2\text{-}CH\bullet]_n\text{-} \rightarrow \text{-}[CH_2\text{-}CH\text{-}O\text{-}EGCG]_n\text{-}$ (Grafted crosslink)

In addition, to the radical sites formed by DCP, EGCG increases the number of crosslinking sites due to the higher number of phenolic groups. We also highlighted this crosslinking mechanism in our previous work on tea polyphenol assisted crosslinking of UHMWPE[39]. The recently reported work by Fu et al.[37,40], also corroborated the results, who concluded that natural polyphenols, gallic acid and dodecyl gallate consisting of three hydroxyl groups individually, resulted in a higher oxidation resistance in crosslink UHMWPE compared to the VE-UH.

In both the EGCG and lsEGCG blended UHMWPE samples, the percent crystallinity is reduced, particularly lsEGCG-UH blend showed a large decrease, which may reflect the creation of a crosslink network structure in comparison to virgin UHMWPE (Figure 3b). The results agree with several previously reported studies[38,41,42], that explain how crystal growth is restrained due to further controlling the chain mobility because of increasing the crosslinked network offered by tea polyphenols blended samples. lsEGCG and EGCG are intended to scavenge surplus radicals that are not taking part in the cross-linking process. Similar to the commonly used antioxidant VE, it has the ability to stabilize residual radicals by providing protons[43]. Interestingly, the melting curve of Virgin UHMWPE and lsEGCG-UH was observed to be wider compared to VE-UH and EGCG-UH, indicating a broad melting range for these polymers (Figure 3a). In contrast, VE-UH and EGCG-UH exhibited a sharper melting curve, suggesting a more well-defined melting behavior. It is also noteworthy that the T_m of VE-UH and lsEGCG-UH were found to be nearly identical, at 149 °C and 148 °C, respectively. This observation suggests that the addition of VE or lsEGCG to UHMWPE has a similar effect on the melting behavior of the resulting polymer[25].

The coefficient of friction (COF) is notably low in the initial few seconds for each of the tested samples and then increase to an equilibrium value (Figures 4a and b). In the beginning, the polymer has a smooth and lubricating surface, keeping the friction coefficient at a minimum. However, as the test continues the surface becomes rough, causing an increase in the friction coefficient until it reaches an equilibrium value[44]. A significant increase was observed in COF values of crosslinked samples relative to uncross linked samples (Figures 4a and b). The direct correlation between the coefficient of friction and crosslink density has been reported in several research studies, which is comparable to our work. As the degree of crosslinking increases, the more tangled structure of the polymer fibrils leads to a decline in the polymer chains on the outer surface, playing a role in brush-mediated lubrication, which results in an increase in the coefficient of friction[45]. The compact crosslinked structure inhibits the motion of the UHMWPE molecular chains, which causes a rise in the shear stress or coefficient of friction in the crosslinked UHMWPE samples. The VE-UH showed a minimum coefficient of friction compared with virgin UHMWPE and tea polyphenol blended UHMWPE. A negligible increase in the coefficient of friction occurs in VE blended UHMWPE after the crosslinking process, which may suggest the crosslinking structure is not quite effective. Contrary to that, EGCG-UH has the highest coefficient of friction before and after crosslinking.

The crosslinking achieved on the surface of lsEGCG-UH and EGCG-UH samples resulted in a wear volume of about 0.086 mm^3 (Figure 5). This wear volume of tea polyphenol is nearly comparable to the wear tests conducted on a similar blended sample (test conditions remain constant), for example, 0.12 mm^3 wear volume for 0.1 and 0.3 wt% EGCG blended UHMWPE[26,46]. Crosslinking brought an apparent decrease in wear volume in four samples. The VE blended UHMWPE resulted in the highest wear volume of 0.13 and 0.092 mm^3 before and after crosslinking respectively, in agreement with the observance of lower crosslinking density in the VE blended UHMWPE. The wear volume of tea polyphenols blended UHMWPE was found to be nearly identical to that of virgin UHMWPE, a result that is consistent with the observed crosslinking density (Figure 2c). The wear results corroborate the direct relation between the crosslinking density and wear resistance, which is closely linked to the failure of joint implants[47,48].

In UHMWPE used for TJR, the interplay between crystallinity, coefficient of friction, and wear resistance is a critical consideration[49]. While crystallinity is traditionally associated with improved mechanical strength due to the presence of highly ordered molecular regions, the current study demonstrates that low crystallinity, when combined with high crosslink density, can also yield favorable tribological performance. Specifically, UHMWPE samples stabilized with tPPs exhibited relatively lower crystallinity (Figure 3b) yet achieved higher crosslink density as a result of surface chemical crosslinking (Figure 2c). These samples showed an increased coefficient of friction, which may be attributed to the reduced chain mobility and enhanced surface stiffness induced by the dense crosslinked network (Figure 4).

The reduced crystallinity in these materials also offers advantages in terms of fatigue resistance and oxidative stability, both crucial for long-term implant durability[50,51]. Excessive crystallinity can lead to embrittlement and crack propagation under cyclic loading, whereas a lower crystalline content helps retain ductility. Post-irradiation thermal treatments like, remelting or annealing, can further optimize this balance by eliminating residual free radicals to mitigate oxidative degradation while permitting partial crystallinity recovery to modulate frictional properties. Thus, while low crystallinity may slightly elevate interfacial friction, the primary wear-reduction mechanism remains the crosslinked microstructure, making such UHMWPE formulations particularly suitable for arthroplasty applications where osteolysis due to wear debris is a predominant concern[52]. The enhanced crosslink density likely acts as a structural reinforcement that stabilizes the amorphous regions and mitigates chain scission and wear particle generation. Therefore, the combination of low crystallinity with high crosslink density offers a promising design strategy for improving the long-term durability and functional performance of UHMWPE in joint replacement applications.

The SEM images of the wear tracks for both crosslinked and un crosslinked UHMWPE samples after wear tests are depicted in Figure 6. It is observed that in the un crosslinked samples the worn surface displays common factors of abrasion and oxidation wear, where a series of grooves, furrows and flakes can be seen along the sliding direction. This suggests that abrasive wear is the leading wear mechanism caused by the loaded ceramic ball[49,53]. Virgin UH had deep and wide grooves and furrows before crosslinking which were only bettered to lines and scratches after surface crosslinking. Flakes and wear particles were observed on the surface of VE-UH blends suggesting the oxidation of the sample. This could be caused by the reaction of un-reacted peroxide free radicals with the atmospheric oxygen hence the surface is oxidized and a slight chunk has been removed[54]. This loss of material and delamination is also known as third body particles or wear debris, and is considered as one of the leading causes of early retrievals in UHMWPE based joint implants[6,55]. On the contrary, there are minimal flakes found on the surface of EGCG-UH and lsEGCG-UH blends, suggesting the higher oxidation resistance of tea polyphenols even under harsh environments[56]. However, there were still few lines and scratches on the surface of un crosslinked EGCG-UH blends which could be linked to its lower surface roughness Figure 2d. After crosslinking lsEGCG-UH blends shows a smother surface without any observable damage. It indicates that highly wear resistant surface is achieved through peroxide crosslinking in the presence of tea polyphenols.

One of the limitations of this work is the chemical analysis of the peroxide and its byproducts in the presence of tea polyphenols. Which could be considered in the future work to observe its effects on the surface oxidation and study the long-term durability under accelerated aging for UHMWPE blends. Nevertheless, these findings underscore the potential of incorporating tea polyphenols into UHMWPE formulations to mitigate wear and enhance durability, offering promising implications for total joint replacements where wear resistance is crucial.

5. Conclusions

A comparative analysis was conducted to assess the effect of surface crosslinking on the overall wear performance of natural antioxidants (VE and tea polyphenol) stabilized UHMWPE blends. The blends were surface crosslinked through the diffusion and decomposition in di-cumyl peroxide followed by comparative testing to evaluate the surface properties such as crosslink density, surface roughness, co-efficient of friction and wear performance in detail. The results yielded three key findings: (1) crosslink density analysis confirmed 17% greater crosslink density in tea polyphenol blends ($p<0.05$), (2) pin-on-disk testing showed 32.6% lower wear rate compared to vitamin E-stabilized which only shows a cumulative decrease of 29.4% in wear volume after surface crosslinking, and (3) surface analysis using SEM revealed ~95% reduced surface damage in lsEGCG-UH and EGCG-UH blends as compared to VE-UH samples. These results demonstrate that tea polyphenol not only enhances crosslinking efficiency during peroxide diffusion but also generates a highly wear-resistant surface architecture. These findings establish tea polyphenol as a superior alternative to vitamin E for developing surface-crosslinked UHMWPE implants, offering both enhanced oxidative stability and wear resistance for prolonged joint replacement longevity.

6. Acknowledgements

The authors greatly acknowledge the financial support of the Higher Education Commission Grant ((Ph-II-MG-5)/PAK-TURK/R&D/HEC/2018) and Pakistan Science Foundation/ National Science Foundation China Grant (PSF/NSFC-II/ ENG/KP-UET (02)).

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Data Availability

The entire dataset supporting the results of this study was published in the article itself.