Improving the Mechanical Properties, Roughness, Thermal Stability, and Contact Angle of the Acrylic Polymer by Graphene and Carbon Fiber Doping for Waterproof Coatings

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Abstract
A polymeric coating was enlarged by incorporating carbon-based nanofiller such as graphene (GR), graphene oxide, chopped carbon fibers (CF), etc., into the polymer to be utilized for various technological applications. In this framework, the acrylic polymer composites and hybrid coatings were made by casting a combination of acrylic polymer, GR, and CF at room temperature. The polymer hybrids were composed of different ratios (0.25, 0.5, 1, and 2 wt%) of GR or (0.25, 0.5, 1, and 2 wt%) of GR and 5 wt% CF. The morphology, thermal stability, glass transition, decomposition temperature, roughness, and the contact angle of the prepared polymer composites and hybrids were investigated. The roughness of the fabric surface of acrylic polymer composites was found to be greatly reduced as the weight ratio of GR or GR + 5 wt% CF was increased. The greatest contact angle was determined to be 83.07° for hybrids containing 2 wt% GR + 5 wt% CF, whereas the least roughness was recorded for composite containing 2 wt% GR and equals 1.22 μm. The addition of CF to polymers composites increased the roughness and contact angle of acrylic polymer composites. The maximum thermal stability was observed for acrylic polymer + 2 wt% GR + 5 wt% CF composites. The maximum value of the impact strength was observed for acrylic polymer hybris containing 1 wt% GR + 5 wt% CF. The Shore A hardness was steadily increased with increasing GR or GR + 5 wt% CF in the hybrid’s polymer. The presence of GR or Gr and CF in the formed hybrids polymers has resulted in an improvement in the mechanical properties, wettability, thermal stability, and hydrophobicity, and hence might be employed for waterproofing coatings.

Keywords Acrylic polymer · Graphene · Carbon fibers · Waterproof · Contact angle · Roughness · Thermal stability · Impact strength · Hardness

1 Introduction
In general, polymeric materials can fail due to several factors such as external weathering, friction, chemical interactions of water, and heat effects, which might occur quickly or over a long period [1–9]. The polymer treatment by surface modification and/or filler loading is thought to be an effective technique to address the aforementioned issue, although problems may occur at any moment during treatment [1].

Polymer composites are one of the most utilized systems in material science, with applications spanning from transportation to aerospace [2, 3], building materials [4, 5], and heat dissipation [6, 7].

Composites are usually prepared by mixing two or more distinct materials to create new substances [6]. The physicochemical, especially the mechanical properties of the new substances are significantly different from the individual primary materials [7–9]. Hybrid materials are composed of organic and synthetic components that are often used in the coatings processes [6–9]. The physical and mechanical properties of hybrids polymer nanocomposites were enhanced by the inclusion of nanoscale inorganic compounds [9].

Carbon-based materials, such as graphene, carbon nanotubes, graphene oxide, reduced graphene oxide, graphene quantum dot, etc., have a vast surface area and are chemically, physically, and thermally stable [10–13]. As a result,
such filler materials have garnered interest for research and use in a variety of applications such as gas sensing, degradation, radiation shielding, etc. [10–13]. In addition, nanographene has been used as a filler to strengthen coated materials as well as to improve the lightness, and the capacity to conduct heat and electricity [14]. Also, CF has been employed to reinforce the polymer, which is used in several applications, notably coating materials, because of its brilliant quality and robust mechanical and thermal properties [6]. The wettability of coating materials is one of the most important elements used to determine the material surface type, which may be divided into two types: flexible liquid materials and firm water-resistant materials. The wettability of a substance can be studied based on the evaluation of the contact angle, for example, when the contact angle is less than 90° or greater than 90°, the paint is hydrophilic or hydrophobic, respectively [15]. Hybrid materials are nanocomposites that contain at least one organic or inorganic ingredient [16]. Hybrid materials may be divided into two major sections due to the nature of the contact, each of which comprises organic and inorganic components. Inorganic materials collaborate with weak hydrogen bonds, such as Van der Waals and hydrogen bonds, and the components are joined by polymeric chemical or anion covalent connections [17]. Hybrid materials are employed in automobiles, building materials, medical supplies, paint materials, and other applications [18]. GR and CF are employed in liquid coatings as fillers to cut costs, increase adhesion, strength, and durability, and kill microorganisms [19, 20]. The addition of graphene/multiwalled carbon nanotube hybrid fillers in polycarbonate/ethylene methyl acrylate nanocomposites improves electromagnetic interference shielding as well as mechanical and electrical conductivity [21, 22]. Such materials have applications in gas sensors, separation membranes, tissue engineering, and drug delivery [23].

The goal of this study is to enhance the contact angle, roughness, thermal stability, and mechanical characteristics of an acrylic polymer by integrating GR or GR, and CF into a flexible acrylic polymer that may be utilized in floor coatings and waterproofing. The research was carried out utilizing a variety of techniques, including field emission scanning electron microscope (FE-SEM), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), contact angle, impact strength, and Shore A hardness.

2 Experimental Details

2.1 Materials

Acrylic polymer, density (1.7 kg/L), was purchased from Al Gurg Forsroc LLC Company, United Arab Emirates. The GR was purchased from Sika Top Seal-107 Elastic Company, produced in the United Arab Emirates, while the chopped CF was purchased from Alper Turkucu at Tapi, Turkey. The GR nanoparticles have platelet morphology, and their surface area equals 120–150 m²/g, while the electric conductivity, thermal conductivity, and thermal expansion equal (10⁷ S.m⁻¹ parallel to surface, 10² S.m⁻¹ perpendiculars to the surface), (300 W/m.K parallel to surface, 6 W/m.K perpendicular to the surface), (4–6 × 10⁻⁶ W/m.K parallel to surface, 0.5–1 × 10⁻⁶ W/m.K perpendicular to the surface), respectively. The CF has a density of 1780 kg/m³, and its length and diameter are 2 mm and 4.2 μm, respectively. Figure 1 depicts the surface morphology of GR nanopowder using atomic force microscopy (AFM), and the particle diameter distribution. It has been observed that the average diameter of GR nanoparticles equals 59.22 nm.

2.2 Preparation of Acrylic Polymer/GR and Acrylic Polymer/GR/CF Hybrids

The acrylic polymer/GR and acrylic polymer/GR/CF hybrid were prepared using the casting technique. The acrylic
polymer solution and GR, and CF filler were made by weighing 100 g in total and then were cast before the drying process for 24 h at room temperature. To achieve a homogeneous polymer composite, GR and CF fillers were dissolved in acetone before being mixed with the acrylic polymer. The ratios of GR equals 0.25, 0.5, 1, 2 wt%, meanwhile, the ratio of CF was kept constant at 5 wt%. Then the proposed ratios of the filler were added to the acrylic polymer solution. The mixture solution was put in a glass tube on a magnetic stirrer at room temperature for 1 h. The weight of the acrylic polymer, GR, and CF in the proposed acrylic polymer/GR/CF hybrids are summarized in Table 1. Figure 2 depicts the optical images of the acrylic polymer/GR composites and acrylic polymer/GR/CF hybrids. The produced acrylic polymer is transparent and white. Except for the pure acrylic polymer sample, all acrylic polymer/GR/CF hybrids were found to be black and opaque. The diameter of the composite disc was ~ 18 cm, and its thickness was ~ 1.4 mm. The dimensions of the composite samples provided in Fig. 2 were chosen to be suitable for the impact strength test.

### 2.3 Contact Angle and Surface Roughness

The contact angle was measured by an optical tensiometer apparatus model Theta Lite at room temperature using a sessile drop. The contact angle of water was measured at 45–60 s intervals on a flat sample surface and the mean of the right and left contact angles were recorded. For a single drop, contact angles were measured at regular intervals and the findings were taken as snap photographs. The surface roughness of samples was measured by a roughness tester (TR100 surface roughness tester). The arithmetic average height ($R_a$) parameter gives a general definition of the height variation of surfaces.

### 2.4 Thermal Stability

The relation between the weight loss, due to dehydration and decomposition processes, and time and temperature are known as thermogravimetric [24]. The thermogravimetric analysis was performed using an SDT Q600 TGA (TA Instruments, Newcastle, DE, USA). A sample of around 5 mg was placed in a regular aluminum pan with a cover. The TGA and DSC were measured at a temperature range of 25–1000 °C with a heating rate of 10 °C/min.

| Sample | Acrylic polymer (g) | GR (g) | CF (g) |
|--------|---------------------|--------|--------|
| Pure acrylic polymer | 100 | 0 | 0 |
| Acrylic polymer + 0.25 wt% GR | 99.75 | 0.25 | 0 |
| Acrylic polymer + 0.5 wt% GR | 99.5 | 0.5 | 0 |
| Acrylic polymer + 1 wt% GR | 99 | 1 | 0 |
| Acrylic polymer + 2 wt% GR | 98 | 2 | 0 |
| Acrylic polymer + 0.25 wt% GR + 5 wt% CF | 94.75 | 0.25 | 5 |
| Acrylic polymer + 0.5 wt% GR + 5 wt% CF | 94.5 | 0.5 | 5 |
| Acrylic polymer + 1 wt% GR + 5 wt% CF | 94 | 1 | 5 |
| Acrylic polymer + 2 wt% GR + 5 wt% CF | 93 | 2 | 5 |

**Fig. 2** Optical images of pure acrylic polymer, acrylic polymer/GR composites, and acrylic polymer/GR/CF hybrids (diameter = 18 cm)
2.5 Surface Morphology

A field emission scanning electron microscope (FE-SEM) model (MIRAIII), a manufacturer firm (TESCAN), and a made nation (Czech Republic) was used to investigate the surface morphology of the polymer composite samples. The polymer composites and hybrids samples were sputter-coated with gold before the SEM examination to avoid charge buildup.

2.6 Mechanical Examinations

The impact strength was determined using the drop tester IDM-P0007 by estimating the minimum height from which the 20 mm diameter impactor falls, producing mechanical damage to the coating. Impact with a 2 kg impactor from a height of 1 m was performed in the test.

The hardness of the cured material was determined using Shore A durometer model HT-6510A. The hardness of the sample was tested more than ten times at various locations on its surface, and the average values were estimated. The softest durometer is penetration, which measures a material's resistance to penetration or scratching. The force that binds the atoms or molecules in the substance determines hardness, which is the softest metric.

3 Results and Discussion

3.1 The Contact Angle of Acrylic Polymer Composite and Hybrids

The wetting behavior of the acrylic polymer/GR/CF hybrids to water has been investigated through the estimation of the contact angle as shown in Fig. 3. The greatest value of the contact angle was estimated to be 83.07° for acrylic polymer hybrids containing 0.25 wt% GR and 5 wt% CF. For acrylic polymer composite containing 1 wt% GR, the contact angle has a minimum value of 48.53°. This is an indicator of the nonpolar surface's attraction to a polar liquid [15]. The inclusion of GR nanofillers/CF makes the acrylic polymer surface more non-polar, i.e., hydrophobic, and increases the contact angle. For acrylic polymer hybrids containing 0.25 wt% GR and 5 wt% CF, the contact angle of acrylic polymer coating with water rises by a factor of 17.3% from 68.74° to 83.07°. Table 2 summarizes the contact angle (θ) for the composites as well as the hybrid’s acrylic polymer on the right and left

Fig. 3 contact angle of a pure acrylic polymer, acrylic polymer composites containing: b 0.25 wt% GR, c 0.5 wt% GR, d 1 wt% GR, e 2 wt% GR, and polymer hybrids conating f 0.25 wt% GR+5 wt% CF, g 0.5 wt% GR+5 wt% CF, h 1 wt% GR+5 wt% CF, and i 2 wt% GR+5 wt% CF
as well as the average contact angle. The concentration and presence of GR and CF in polymer composites and hybrids have a substantial effect on the wetting surface, which is consistent with previous research [25].

3.2 Surface Roughness of Acrylic Polymer Composite and Hybrids

Figure 4 depicts the roughness behavior of pure acrylic polymers, acrylic polymer/GR, and acrylic polymer/GR/CF hybrids. The figure depicts changes in the arithmetic average height (Ra) values versus the concentration of GR. The surface roughness of the acrylic polymer demonstrates that the fabric surface roughness values dropped dramatically with increasing the GR and CF contents, which agrees with ref. [26]. The deduced roughness for pure acrylic polymer and acrylic polymer composites contains 2 wt% GR equals 2.73 and 1.22 μm, respectively. The addition of CF filler to acrylic polymer/GR composite was raising the surface roughness. The maximum surface roughness value was obtained for acrylic polymer hybrids containing 0.25 wt% GR + 5 wt% CF which was caused by height distribution discrepancies between the yarn crown peaks of fibers and gaps caused by the junction of the CF on the sample’s surface.

3.3 Thermal Stability of Acrylic Polymer Composite and Hybrids

TGA can provide information on chemical events such as dryness, dehydration, decomposition, oxidation, and reduction. Besides, the TGA provides information about some transition and transformation processes like as second-order phase transitions, which include vaporization, sublimation, absorption, and desorption. Furthermore, the TGA is utilized to assess the thermal stability of polymers [27]. Figure 5 shows the TGA curves of acrylic polymer/GR composites and acrylic polymer/GR/CF hybrids. In general, all TGA curves exhibit a one-step degradation pathway which means the addition of GR and CF fillers didn’t affect the matrix polymers’ degradation mechanism. The first degradation occurred at 450 °C for all acrylic polymer composites and hybrids. The results show that the polymer hybrids become more thermally stable and linked to the GR and CF concentration. The weight loss of the composites and hybrids was reduced as the GR and CF content was increased, which is consistent with ref. [28]. The weight loss at 450 °C, total weight loss at 1000 °C, glass transition temperature (T_g), and decomposition temperature (T_d) for different acrylic polymer composites and hybrids are shown in Table 3. At 450 °C,

| Sample                        | Θ_1     | Θ_2     | Θ  |
|-------------------------------|---------|---------|----|
| Pure acrylic polymer          | 69.66   | 67.82   | 68.74 |
| Acrylic polymer + 0.25 wt% GR | 58.08   | 55.62   | 56.85 |
| Acrylic polymer + 0.5 wt% GR  | 67.43   | 66.50   | 66.96 |
| Acrylic polymer + 1 wt% GR    | 50.09   | 46.98   | 48.53 |
| Acrylic polymer + 2 wt% GR    | 64.13   | 61.89   | 63.01 |
| Acrylic polymer + 0.25 wt% GR + 5 wt% CF | 83.10 | 83.04 | 83.07 |
| Acrylic polymer + 0.5 wt% GR + 5 wt% CF | 75.85 | 76.51 | 76.18 |
| Acrylic polymer + 1 wt% GR + 5 wt% CF | 66.63 | 69.64 | 68.13 |
| Acrylic polymer + 2 wt% GR + 5 wt% CF | 65.70 | 65.57 | 65.63 |

Table 2 left contact angle (Θ_1), right contact angle (Θ_2), and average contact angle (Θ) of acrylic polymer/GR composites and hybrid acrylic polymer/GR/CF.
the minimum and maximum weight losses are 38.5% and 49.9% for acrylic polymers hybrids containing 2 wt% GR + 5 wt% CF and acrylic polymers composites containing 2 wt% GR, respectively. On the other hand, at 1000 °C, the minimum and maximum total weight losses are 84.9% and 100%, respectively, for acrylic polymer hybrids containing 2 wt% GR + 5 wt% CF and pure acrylic polymer. From the above discussion, the acrylic polymer hybrids containing 2 wt% GR + 5 wt% CF exhibit greater thermal stability than the other acrylic polymer composites and hybrids.

Fig. 6 depicts the DSC curves for acrylic polymer composites and hybrids containing various ratios of GR and CF performed in the temperature range of 25–1000 °C. The glass temperature is shown by one vertex in the DSC curve. The DSC traces were used to determine the glass transition and decomposition temperatures, which are given in Table 3. The investigated acrylic polymer composites and hybrids showed a decrease in $T_g$ value with increasing GR and CF filler content. The highest glass transition temperature and maximum decomposition temperature were measured for acrylic polymer hybrids loaded with 2 wt% GR + 5 wt% CF. This observation could be attributed to the less interaction between the polymer mixture and filler molecules, hence resulting in a free surface. In addition, when the filler content was increased, the free volume was increased and hence resulting in a drop in $T_g$. This behavior reveals better thermal stability of acrylic polymer hybrids containing GR and CF compared to the pure acrylic polymer. The inclusion of GR and CF inside an acrylic polymer matrix has traditionally been claimed to have superior thermal stability.

### Table 3

| Sample                              | Weight loss (%) at 450 °C | Total weight loss (%) at 1000 °C | $T_g$ (°C) | $T_d$ (°C) |
|-------------------------------------|---------------------------|---------------------------------|------------|------------|
| Pure acrylic polymer                | 49.6                      | 100                             | 172.4      | 419.3      |
| Acrylic polymer + 0.25 wt% GR       | 49.3                      | 97.6                            | 186.5      | 404.7      |
| Acrylic polymer + 0.25 wt% GR + 5 wt% CF | 44.5                      | 91.9                            | 228.0      | 418.8      |
| Acrylic polymer + 2wt% GR           | 49.9                      | 97.7                            | 189.4      | 420.8      |
| Acrylic polymer + 2wt% GR + 5 wt% CF | 38.5                      | 84.93                           | 231.4      | 421.2      |

Figure 6 depicts the DSC traces (temperature difference (∆T) versus temperature) for different samples of acrylic polymer/GR composites and acrylic polymer/GR/CF hybrids.

3.4 Morphology of Acrylic Polymer Composite and Hybrids

Figure 7 shows the selected SEM images of acrylic polymer composites doped with 0.25 wt% GR, and acrylic polymer hybrids doped with 0.25 wt% GR + 5 wt% CF. The SEM images for acrylic polymer composites containing 0.25 wt% GR reveal the presence of small spherical particles inside the matrix with a homogenous distribution. The observed spherical particles belong to the GR nanoparticles inside the polymer matrix. The average particle size of the GR nonportable was estimated to be 28.5 nm. On the other side, the SEM images for acrylic polymer hybrids doped with 0.25 wt% GR + 5 wt% CF reveals the presence of platelet shape besides the spherical nanoparticles in the polymer matrix. The observed platelet shapes could belong to the CF, and their average estimated width was closed to 75.1 nm. The polymer composite was made up of an acrylic polymer matrix and the filler’s reinforcing phase of GR and CF [29].

3.5 Mechanical Properties of Acrylic Polymer Composite and Hybrids

Figure 8a shows the impact strength of acrylic polymer composites and hybrids. It was observed the impact strength of acrylic polymer composite was increased as the weight ratio of GR was increased. It is worth noting, that the change in the impact strength of acrylic polymer hybrids shows different behaviors compared to acrylic polymer composite. It was observed that the impact strength was increased as the GR ratio was increased up to 1 wt%, and then was decreased for a further increase in the GR ratio. The maximal impact
strength was reported for acrylic polymer hybrids comprising 1 wt% GR and 5 wt% CF and reached 26.5 J/m. The decrease in the impact strength for a further increase in the GR content may be attributed to the geometry (greater surface area) of GR nanoparticles which promotes effective matrix-reinforcement bonding. The ability of GR and CF as reinforcing materials was attributed to the presence of hydroxyl and acrylic polymer residue groups on their surfaces [30]. Micromechanical interlocking and covalent bonding with acrylic polymer matrices increase the interstitial adhesion between matrix and fiber, resulting in the higher mechanical efficiency of GR and CF-containing hybrids [30]. It was deduced that GR and CF together have a greater influence on the impact strength of the acrylic polymer compared to GR doping only.

For the slurry matrix polymer film, the presence of a fine space structure of GR and CF leads to a poor deformation ability of the acrylic polymer meanwhile improving the micro-structure of the acrylic polymer. Figure 8b shows the Shore A hardness for acrylic polymer composites and hybrids. It was seen from the figure an improvement in the hardness of the acrylic polymer composites and hybrids with increasing the GR ratio. Such improvement resulted from the super interaction between GR and the polymer matrix. In addition, the improvement in the hardness can be attributed to the increase in the surface area of the filler and excellent filler dispersion. A considerable improvement in the hardness was found for acrylic polymer hybrids containing 1 wt% GR and 5 wt% CF. The mechanical characteristics of polymer composites can be influenced by a variety of parameters, including particle
distribution and loading, surface adhesion between the matrix and filler particles, filler particle size, and formation [31]. Increasing the weight ratio of GR to 2 wt% leads to a decrease in hardness values due to the accumulation of GR particles and hence resulted in a decrease in the surface area of the accumulated GR particles, thus reducing adhesion and forming a separate phase in the polymer matrix.

4 Conclusion

In summary, the casting approach effectively produced acrylic polymer/GR composites and acrylic polymer/GR/CF hybrids. The concentration of GR influences the contact angle of acrylic polymer composites and hybrids. The introduction of CF enhances contact angle, with a maximum contact angle of 83.1° reported for acrylic polymer/GR/CF hybrids comprising 0.25 wt% GR, resulting in a non-wetting surface. Acrylic polymer/GR/CF hybrids having 0.25 wt% GR has a high surface roughness rating, which decreases as the GR weight ratio increases. The thermal stability and glass transition temperature of acrylic polymer composites and hybrids are higher than that of the pure acrylic polymer and can be improved as the GR content increases. The GR and CF fillers were homogeneously dispersed throughout the acrylic polymer composites and hybrids and have spherical and platelet forms inside the polymer matrix, respectively. Shore A hardness and impact strength of acrylic polymer hybrids have substantially higher than pure acrylic polymer and acrylic polymer/GR composites. The Shore A hardness rose as the GR weight ratio increased and a significant improvement was seen, notably for polymer composites comprising 1 wt% GR + 5 wt% CF. Generally, compared to pure acrylic polymer and acrylic polymer/GR composites, the physical and mechanical characteristics of the produced acrylic polymer hybrids are enhanced. The generated acrylic polymer composites and hybrids might be used in a variety of applications, including waterproofing coatings.

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