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Interfacial adhesion evaluation *via* wettability for fiber reinforced polymer composites: A review

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ABSTRACT

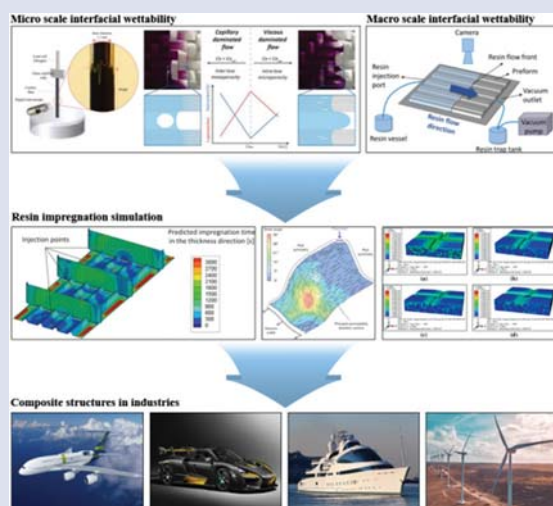
As the demand for fiber-reinforced composite (FRC) has increased in various industries, composite materials have been manufactured in larger sizes and more complex shapes. Since the FRC has been manufactured in such larger and more complex shapes, wettability, one of the important factors in FRC manufacturing efficiency, has been the focus of many researchers. This paper explores various evaluation methods of the wettability between fiber and polymer matrix. Generally, work of adhesion, capillary, and permeability methods have been used to evaluate the wettability parameters between the fibers and the polymer matrix. These three parameters exhibit different scales of measurements such as surface energy, viscosity of the polymer, the fiber volume fraction, fiber orientation, and so on. Future research may include complementary studies between these evaluation methods.




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1. Introduction

1.1. The importance of wettability in fiber-reinforced composite

Due to their lightweight and good mechanical properties, application of fiber-reinforced composites (FRC) has been tried as structural materials in many industries [1,2]. FRC materials have been developed and used for a wide range of applications in aircraft, ships, automobiles, wind turbine blades, and so on [3,4]. As the demand for FRC has increased in various industries, FRC materials are being manufactured more quickly and elaborately with near-zero defect rates [5,6]. Wettability is one factor used to evaluate manufacturing efficiency, and interfacial properties can be evaluated by this factor in a roundabout way [7–9].

To improve the manufacturing efficiency of composite materials, the wettability, between fiber and polymer resin, should be improved. In the case of poor wettability, the polymer matrix does not fully impregnate the fibers [10–12]. This can lead to micro voids, dry zones, and poor reinforcement [13,14]. Poor wettability also led to low mechanical properties of FRC due to poor interfacial properties. Load transfer from fiber reinforcement to polymer matrix is dominated by the interface between fiber and matrix. Good interfacial properties reduce local stress concentrations and internal crack propagation thereby increasing the mechanical performance of the composite [15–17].

The wettability between fiber and polymer matrix has been evaluated using various evaluation methods. In general, work of adhesion [18–20], capillary action [21–23], and permeability [24–26] are used to evaluate wettability, under different material conditions, to optimize the manufacturing efficiency of the FRC. Three parameters were calculated under different material conditions, and previous publications have studied how modifying the equations of wettability with these different conditions can improve wettability.

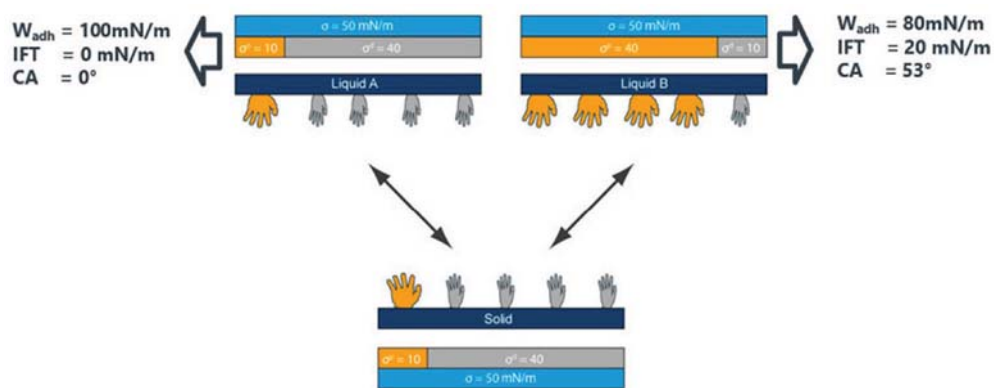


Figure 1. Schematic arrangement of work of adhesion with polar and disperse components [27].

2. Experimental

2.1. Surface energies and work of adhesion

Surface energy and work of adhesion have been used to predict wettability between two different materials. Figure 1 shows two liquids with the same surface tension but opposite polar and disperse component ratios. The polar and disperse components of both the liquid and substrate are designated as the yellow and gray bands, respectively. When the liquid and solid come into contact, the liquid and the substrate with the highest interaction (liquid A) show the highest work of adhesion and the lowest interfacial tension. This suggests that liquid A and substrate combination will have a better initial adhesion and, over time, will be more stable than liquid B. Liquid B has a minimal interaction with the substrate, lower work of adhesion and higher interfacial tension [27–29].

In FRC, the wettability between fiber and polymer resin was determined using the work of adhesion, W_a . The contact angles (CA) of fiber and polymer resin were measured using four different solvents (i.e., distilled water, formamide, ethylene glycol, and diiodomethane) for static and dynamic CAs. The contact angles were calculated using Young's equation [30]:

$$\gamma_s - \gamma_{SL} = \gamma_L \cos \theta \quad (1)$$

where γ_L , γ_{SL} , and γ_s are the liquid surface tension, the solid/liquid interfacial energy, and the solid surface energy, respectively. The total surface energy, γ^T , is the sum of the Lifshitz-van der Waals component, γ^{LW} and the acid-base component, γ^{AB} . The calculation of these components followed the modified young-Dupre equation [31] of the work of adhesion, W_a expressed as:

$$W_a = \gamma_L(1 + \cos \theta) = 2(\gamma_L^{LW} \gamma_s^{LW})^{\frac{1}{2}} + 2\left[(\gamma_s^- \gamma_L^+)^{\frac{1}{2}} + (\gamma_s^+ \gamma_L^-)^{\frac{1}{2}}\right] \quad (2)$$

A commonly used approach, in considering solid surface energies, is to express them as the sum of dispersive and polar components which influences the work of adhesion, W_a , between the surface of the reinforcement material and the matrix. To determine the polar and dispersive surface free energies, the Owens–Wendt equation [32] is used, expressed as:

$$W_a = \gamma_L(1 + \cos \theta) = 2(\gamma_s^d \gamma_L^d)^{\frac{1}{2}} + 2(\gamma_s^p \gamma_L^p)^{\frac{1}{2}} \quad (3)$$

where γ_L , γ_L^d , and γ_L^p are known for the testing liquids and γ_s^d and γ_s^p can be calculated from the measured contact angles. Based on the surface energy of the material, the work of adhesion between the fiber and polymer resin was obtained predicting an improvement in interfacial adhesion [33–35].

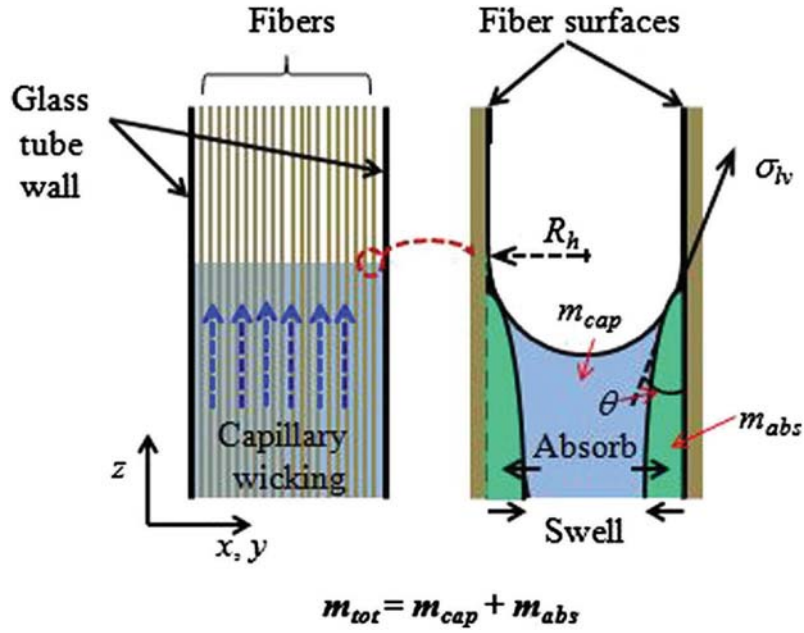


Figure 2. Schematic arrangement of the capillary test [21].

2.2. Capillary

Capillary action is defined as the movement of fluid within the spaces of a porous material due to the intermolecular forces between the liquid and the surrounding solid surfaces that enable the liquid to flow into narrow spaces without the assistance of, or even in opposition to, external forces like gravity [36–38]. In composite materials, this action can be shown by resin impregnation on a micro scale.

In Figure 2, a capillary test method was suggested, using a schematic arrangement. A glass capillary tube with a fiber tow just touched the surface of the polymer causing the resin to impregnate the fiber tow. The variation in weight of the specimens was measured

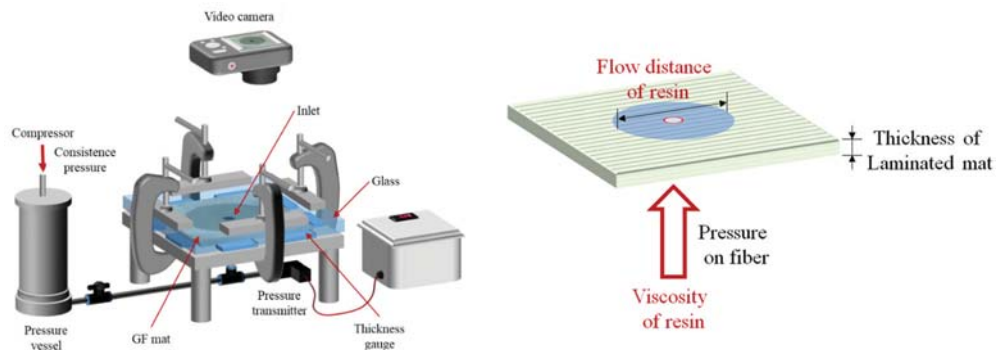


Figure 3. Schematic arrangement for permeability measurement of fiber mat by the polymer resin [24].

and the experimental height of resin impregnation was observed in situ, using a USB microscope. The height of resin impregnation was compared with the calculated height, using Washburn's equation [39]:

$$h^2(t) = \left\{ \frac{(cr)}{2} \right\} \frac{\gamma_L \cos \theta_a}{\eta} t \quad (4)$$

where h is the height of resin impregnation, t is the time, and c is a parameter considering tortuosity. r is the mean porous radius, θ_a is the apparent advancing CA, γ_L is the liquid surface tension of the epoxy resin, and η is the viscosity of the resin.

2.3. Permeability

For FRC, permeability is the resin impregnation factor into a fiber mat on a macro scale. It is a key parameter in liquid composite molding processes; it is a well-defined concept, but still a difficult parameter to quantify, in large part because textile reinforcements are not constant in their internal geometry [40,41]. Permeability is affected by the density and surface area of reinforcing material, the viscosity of the resin, the design of the mold, and so on. A schematic plot of this permeability measurement is shown in Figure 3 and equation (5) relates permeability to the various resin and mat parameters [42]:

$$K_f = -q_f \frac{\mu \cdot h_f}{P_f} \quad (5)$$

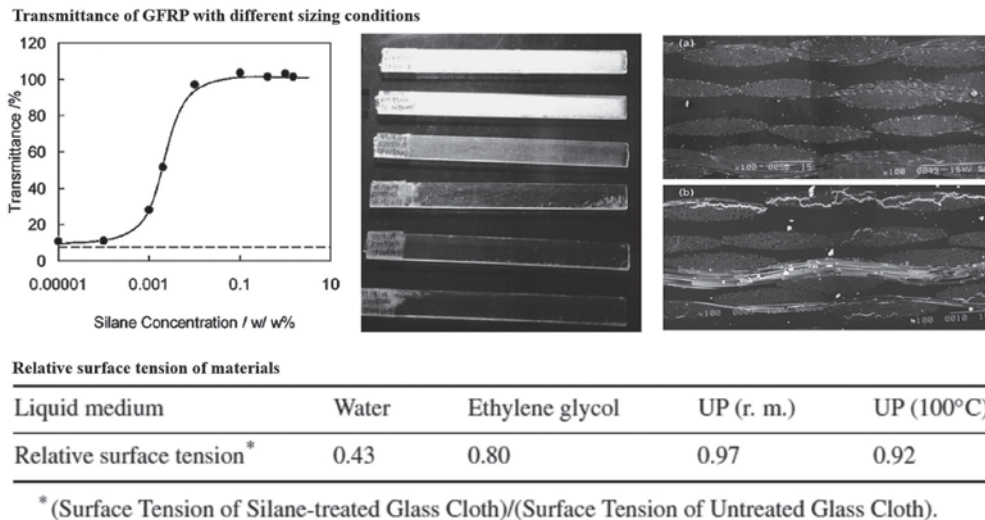
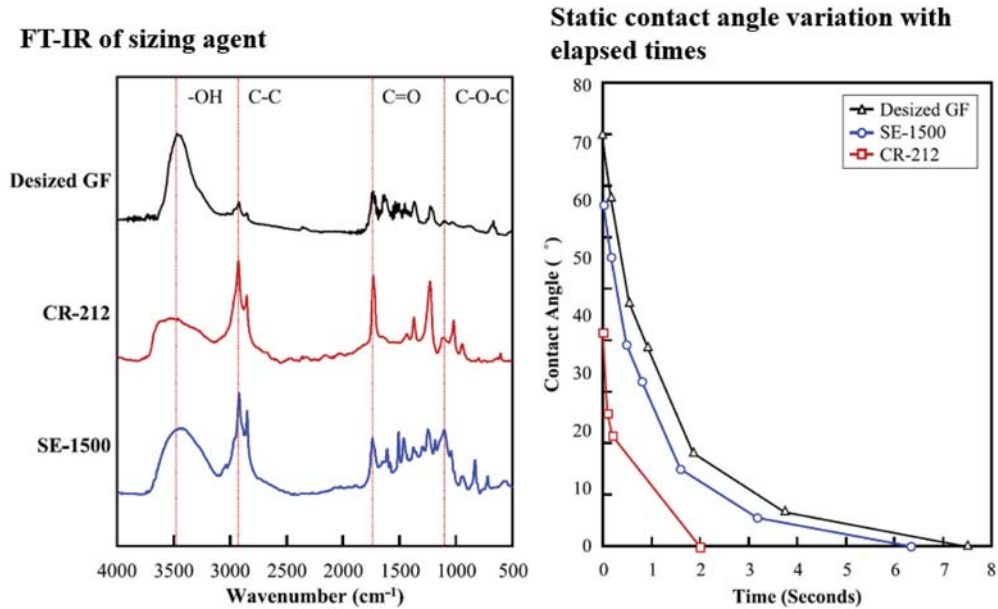


Figure 4. Transmittance of GFRP and relative surface tension of materials with different silane concentrations [12].

where, K_f is the permeability of the fiber mat, q_f is the flow distance of the resin into the fiber mat, μ is the viscosity of the resin, h_f is the thickness of the laminated FRC and P_f is the pressure on the fiber mat. The fiber volume fraction (FVF) was determined using equation (6) and was kept constant for all the permeability measurements.

$$FVF = \frac{FAW \cdot N_f}{\rho_f \cdot h_f} \quad (6)$$

where FAW is fiber area weight, N_f is number layers in the fabric mats, ρ_f is the density of the fiber, and h_f is the thickness of the FRC.



Surface energies and work of adhesion of materials

	γ_s	γ_s^{LW}	γ_s^{AB}	γ_s^+	γ_s^-	γ^d	γ^p	W_a
DCPD	24.9	19.6	5.2	17.8	0.4	10.1	8.6	—
Desized GF	32.6	25.8	6.9	36.6	0.3	12.0	29.3	57.3
SE-1500	34.2	25.5	8.7	22.1	0.8	15.5	21.0	58.4
CR-212	38.1	35.4	2.7	4.2	0.4	32.4	4.6	60.8

Figure 5. Surface energy and work of adhesion between glass fiber and *p*-DCPD for different sizing agent treatments [46].

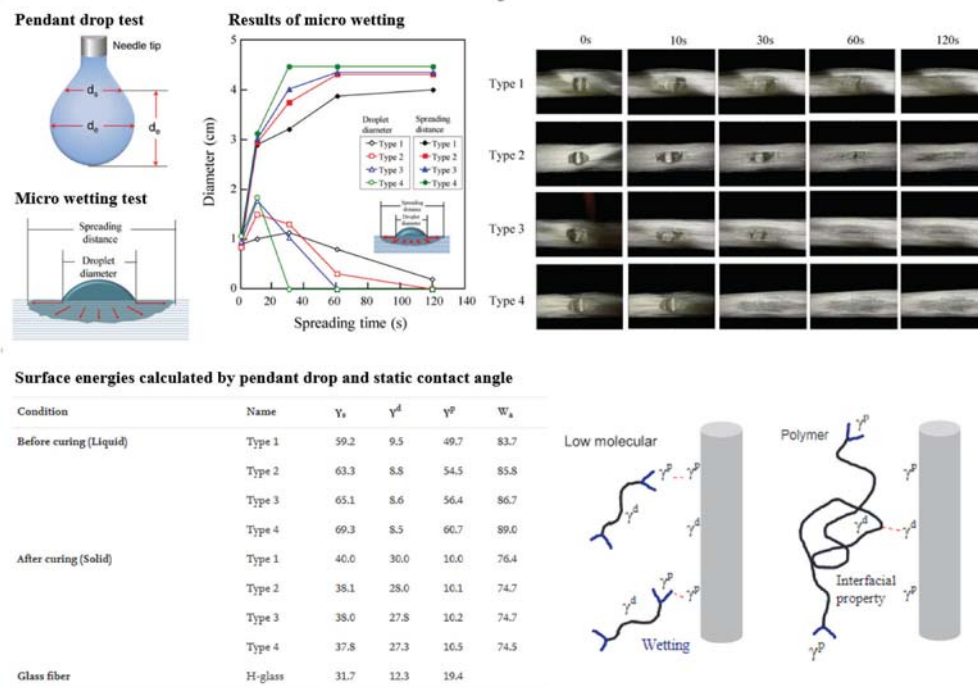


Figure 6. Surface energy and work of adhesion between glass fiber and epoxy resin with different epoxy resin formulations and curing state [51].

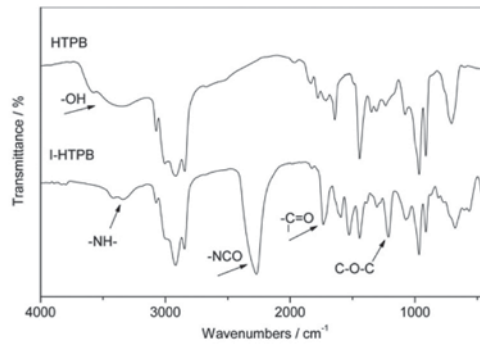
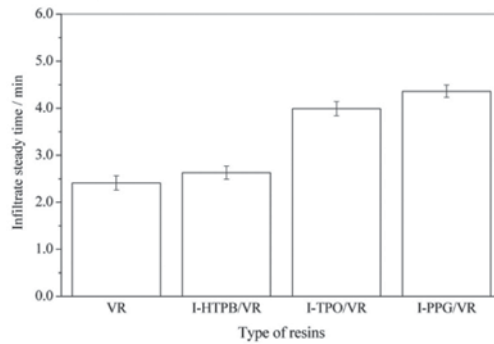
3. Research trend of wettability in fiber-reinforced composites

3.1. Evaluation of wettability using surface energies and work of adhesion

The surface energies and work of adhesion between fiber and polymer matrix are defined as the reversible thermodynamic work required to separate the interface from the equilibrium state of two phases to an infinite separation distance [43–45]. The determination of these parameters is one of the classical evaluation methods for wettability in thermodynamic aspects. The research of Ohnishi *et al.* [12] focused on the wettability between glass fiber (GF) and unsaturated polyester (UP) with different silane concentrations using transmittance of composite and relative surface tension between GF and UP. As shown in Figure 4, the transmittance of the composite was improved as the silane concentration increased to 0.1 wt%. In the FE-SEM photos, the UP resin could not be impregnated well into GF in the case of low silane concentration. The light, which was passed through the composite, was scattered by voids, and the transmittance was reduced. The wettability between GF and UP was evaluated using relative surface tension. As the silane was treated on the GF surface, the relative surface tension exhibited below 1 in all of the solvent and UP resin cases as shown in the summarized table. Using this table, the wettability of UP resin was improved as the silane treatment on GF surface.

Mechanical properties of fiber reinforced composite with different resin conditions

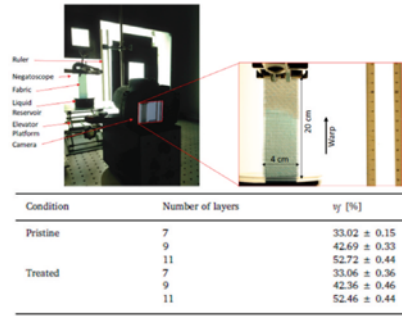
Type of resin	Tensile property		Impact strength (KJ/m ²)	Flexural modulus (GPa)
	Tensile strength (MPa)	Elongation at break (%)		
Original VR	81.95	5.34	2.68	3.37
LNBR/VR	59.47	6.89	4.22	3.08
PIB/VR	53.37	6.56	5.13	3.02
I-HTPB/VR	47.38	7.05	3.60	2.16
I-TPO/VR	8.50	18.20	8.29	0.25

FT-IR of resin with different resin conditions**Capillary test with different resin conditions****Figure 7.** Surface energy and work of adhesion between glass fiber and epoxy resin with different epoxy resin formulations and curing state [58].

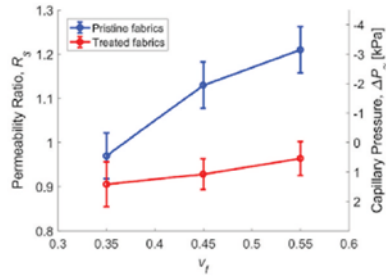
The research of J.H. Kim *et al.* [46] focused on the wettability between GF and *p*-dicyclopentadiene (*p*-DCPD) used surface energies and work of adhesion for different sizing agent conditions. As shown in Figure 5, as the dispersion component of surface energies increased, the work of adhesion also increased for different hydroxyl group amounts, indicated by FT-IR peaks. Static CA measurements were performed to determine the wettability compared to the work of adhesion. As the work of adhesion increased, the decreasing rate of static CA for *p*-DCPD was accelerated. This paper will demonstrate that the wettability of *p*-DCPD resin into GF, was improved as the hydroxyl group of sizing agent decreased [47,48].

Static and dynamic CA are usually used to calculate surface energies of fiber and polymer matrix in the solid state. In the case of the polymer matrix, however, some polymer matrixes, such as epoxy and unsaturated polyester, have been used in uncured states to impregnate the polymer resin into the fibers. In this state, surface energies might differ from those in the cured states [49,50]. To resolve this issue, Shin *et al.* [51] calculated the surface energies using the pendant drop test and compared the results with those of the micro wetting test for different epoxy formulations, with the results shown in Figure 6. The un-cured epoxy resins have a low molecular weight containing a number of functional groups. Functional groups pose the potential for polar interactions [52,53]. This led to a higher work of adhesion than that of cured epoxy. Long chains result in a more intermittent dispersive interaction [54,55]. This dispersive interaction produces bonding between the glass fiber and the epoxy resins as well as improved interfacial properties.

Capillary test equipment and test conditions



Capillary pressure with different fiber volume fractions



Observation of flow front during VARTM in-situ

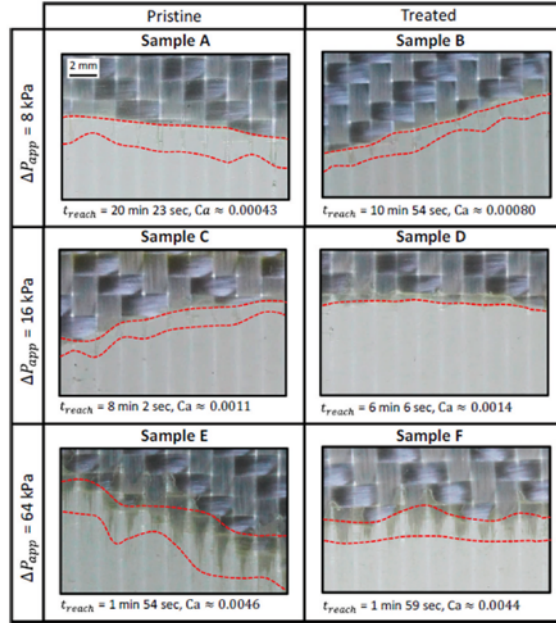


Figure 8. Capillary testing and flow front observation for different FVF and surface treatments [59].

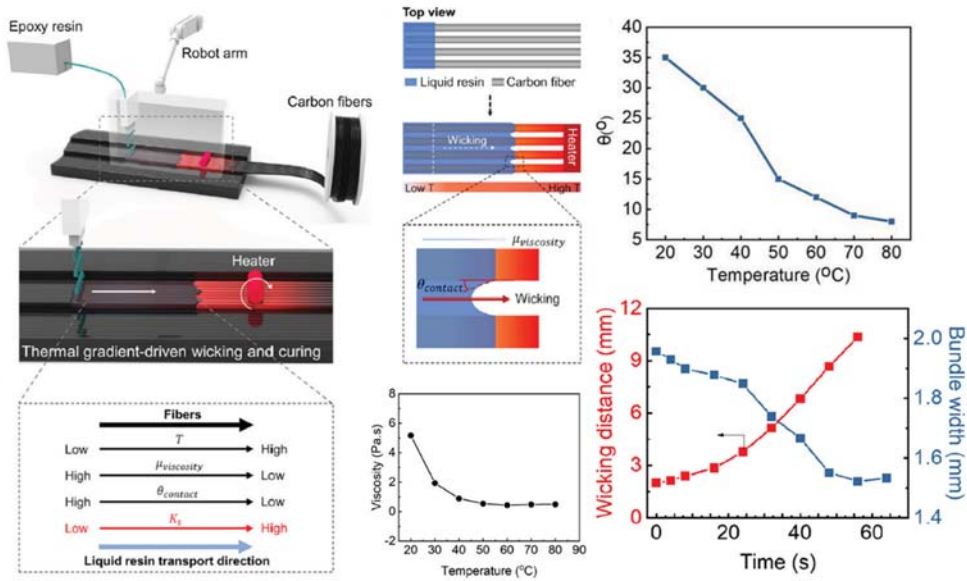


Figure 9. Capillary tests on carbon fibers and epoxy resin at different temperatures [65].

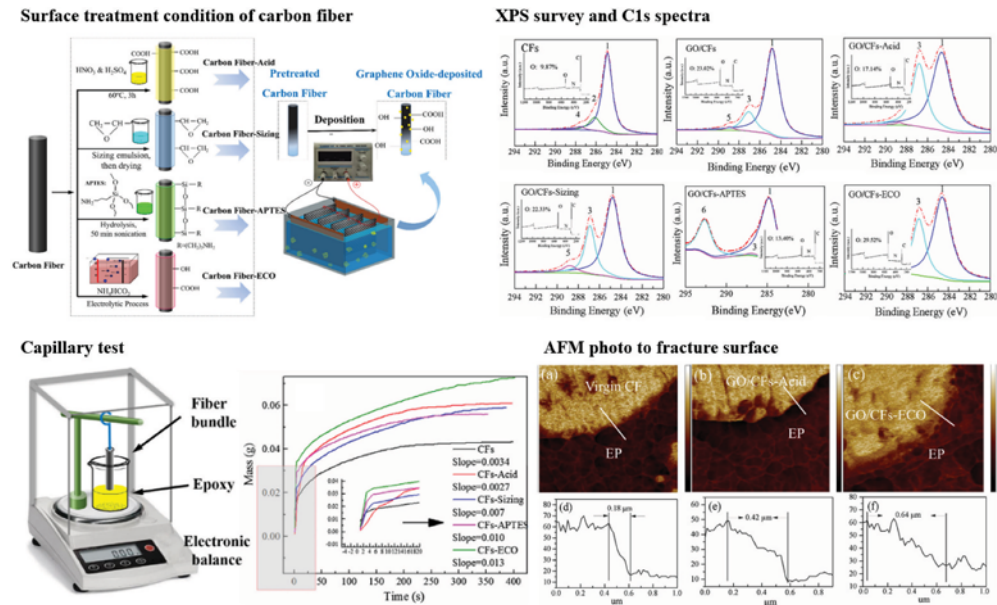


Figure 10. Capillary test and interface observation of carbon fiber and epoxy resin with different surface treatments [68].

3.2. Evaluation of wettability using capillary parameter

The capillary parameter is affected by material conditions and surface energies between fiber and polymer resin [56,57]. The capillary evaluation could be determined more quantitatively than the work of adhesion in micro-scale and material engineering aspects. The research of Zheng *et al.* [58] focused on the wetting behavior of vinyl ester resin (VR) into ultrahigh molecular weight polyester (UHMWPE) fiber with different resin conditions using capillary test. In this publication (Figure 7), many liquid rubbers were added to improve wettability such as liquid nitrile-butadiene rubber (LNBR), liquid polyisobutylene (PBR), hydroxyl-terminated liquid polybutadiene rubber (HTPB), and polyether triols (TPO). As the rubber materials were added into VR, the impact property was improved, while tensile strength decreased. To analyze chemical groups of VR, the FT-IR peak was determined, and there existed many functional groups in VR that could help to improve the wettability by hydrogen bonding such as the hydroxyl, cyano, and etc. As shown in capillary data, the wettability of VR was improved in the rubber materials added cases.

Caglar *et al.* [59] studied the capillary parameter contribution in fabrics with different fiber volume fractions and surface treatments. The fiber volume fraction is one of the main factors controlling the mechanical properties of FRC [60–62]. In Figure 8, as the fiber volume fraction increased, the impregnation of the polymer resin decreased, while the mechanical properties improved. As shown in the photos of the flow front, the resin flow front was moved further forward than the full-impregnation front as the fiber volume fraction decreased. In the resin transfer

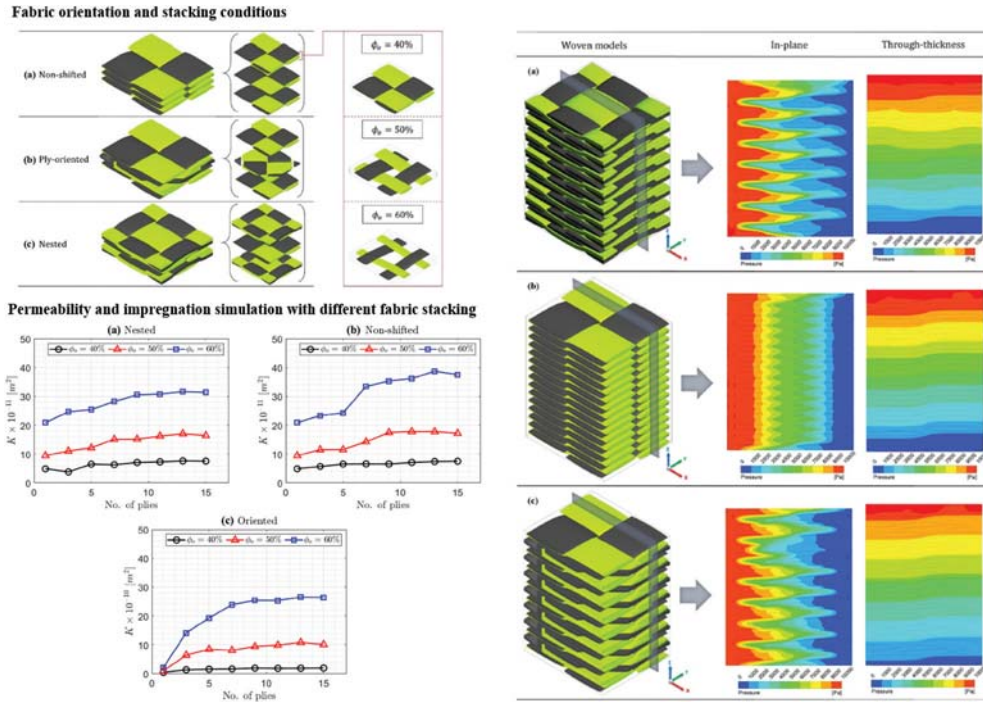


Figure 11. Permeability and impregnation simulation with different fabric orientations and stacking conditions [74].

molding (RTM) process, the resin flow progression is determined by the flow front, and it leads to a more stable and quicker resin flow. It may prevent micro-void formation in the composite and dry zone [63,64].

Figure 9 shows the results of research by B. Shi *et al.* [65] on carbon fiber reinforced epoxy composites, using the viscosity effects of the capillary parameter for 3D printer filaments. The viscosity of the epoxy resin was controlled using a Joule-Heater. As the temperature of the resin increased, as a result of increasing the carbon fiber temperature, the resin viscosity decreased dramatically. The contact angle in the meniscus also decreased as a result of the decreasing viscosity. This was influenced by the more active chain mobility, leading to improved work of adhesion between carbon fiber and resin and the improved impregnation of the resin into the carbon fibers [66,67].

The capillary parameter also influences the surface conditions of the fibers, and it is a factor affecting the contact angle in Darcy's law. Figure 10 shows research results of X. Yuan *et al.* [68] on the wettability of graphene oxide treated carbon fiber with different treatment methods. The surface was analyzed, and the elemental composition of oxygen was determined. Using the data from this analysis, the wettability of the fibers was determined. The wettability was evaluated using the capillary test, and the wettability was improved as the functional group content increased, which included oxygen. The fracture area was observed, using AFM analysis focused on the interface between the fiber and the epoxy matrix. The

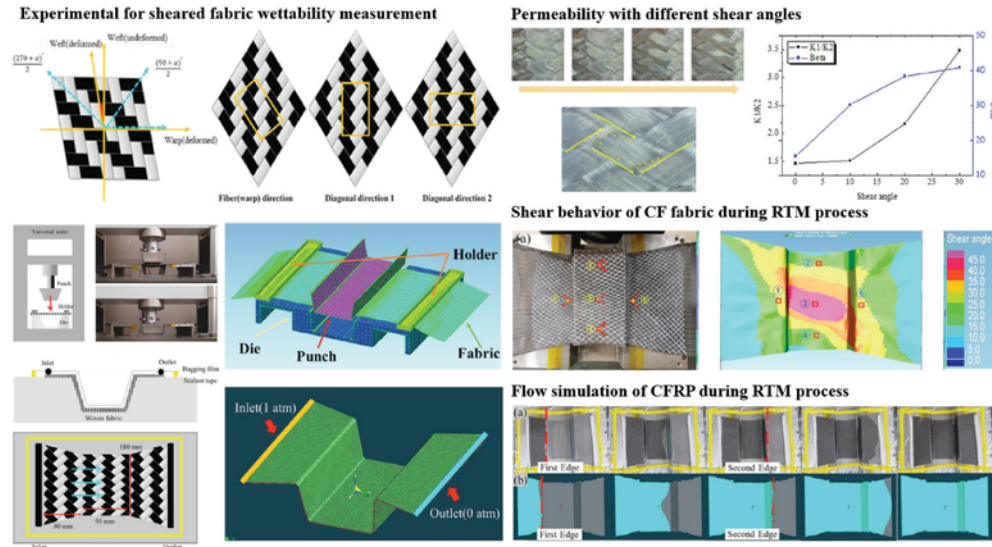


Figure 12. Permeability and impregnation simulation for carbon fibers and epoxy resin with different shear angles of carbon fabric [82].

surface roughness gradually decreased as the functional group content, which included oxygen, was increased. As shown in these results, the interfacial properties were also improved as the oxygen included functional group content increased [69,70].

3.3. Evaluation of wettability using permeability parameter

The permeability is affected by the bulk conditions of a material and manufacturing processing, and their parameters can be determined more quantitatively than work of adhesion in macro-scale in mechanical engineering aspects [71–73]. This parameter can be used for the simulation of resin impregnation for RTM process as well. Alotaibi *et al.* [74] investigated the permeability of fiber reinforced polymer composites with different fiber orientations and laminate stackings. Some of their results are shown in Figure 11. The flow behavior for in-plane and through-thickness was evaluated using permeability for different fiber orientations and laminate stackings. Using these data, the flow behaviors could be visualized using pressure gradient contours. As the fiber orientation became more complex, the flow of the resin in the through-thickness direction exhibited increased flow resistance, which could lead to void formation [75,76]. In this paper, it was found that an evaluation of the dual-scale void during the resin impregnation was important, and it needed to be predicted to prevent failures such as micro-voids and dry zone.

In the real RTM process, the fabric arrangement cannot be controlled homogeneously by shear in the curved edge [77–79]. Fabric arrangement affects resin flow due to the changing fiber volume fraction in specific areas [80,81]. Figure 12 shows results of some of the research of Kim *et al.* [82] in which the permeability of composites was calculated as the fabric was deformed by shear, and the resin flow in the RTM process for complex shapes was predicted with simulation applied with the shear parameter of fabric. The permeability was calculated

for different fabric shear angles, and the aspect ratio of permeability increased dynamically as the shear angle increased. This result was applied to a practical simulation system for resin flow prediction. In the punched section, the fabric was deformed dramatically due to shear behavior by tensile stress [83,84]. The flow during RTM was simulated based on permeability with different shear angles of fabric, and compared with the flow behavior of resin during real RTM processes. As shown in the figures, the resin flow was delayed in sections of deformed fabric. In the RTM process, The behavior of the resin flow front exhibited similarly compared with the simulation.

4. Conclusions

FRC is used in various industries to improve the body weight of structures. As the size and shape of FRC are bigger and more complex in these structural materials, it is important that we have knowledge of wettability in order to find the optimized manufacturing methods and efficiency for FRC. This paper summarizes, from previous publications, the current trends of evaluation methods for wettability, with different scales and material conditions. Micro-scale evaluation methods affect basic material conditions such as surface energies of fiber and polymer resin, formulation of resin, and so on. On a macro-scale, the bulk conditions to manufacture FRC are influenced by wettability such as fiber volume fractions, injection pressure, fabric orientation, and so on. In the future research, studies will be conducted on how these parameters complement each other, and how wettability might be improved to more efficiently and accurately manufacture larger and more complex FRC.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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