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Mussel Inspired Modification of Rubber Crumbs for Improved Interfacial Adhesion in Rubber Cement Mortar

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Abstract

Crumb rubbers exhibit extensive potential applications as infrastructure materials due to the low elastic modulus. Nevertheless, the poor interfacial adhesion between rubber crumb and cement matrix limits the scale applications of crumb rubbers in cement-based composites. In this study, mussel-inspired modification of crumb rubbers is investigated. The hydrophilicity of rubber surface has apparently improved after polydopamine (PDA) modification. Effects of the surface modifications of rubbers on the compressive strength, fluidity, and tribology behaviors of rubberized mortars have been systematically characterized. The superiority of PDA modification for crumb rubbers has been demonstrated by comparing those with the other polyphenol modifications and the routine oxidation modification. The compressive strength of the PDA modified rubber cement mortar increases by 37% comparing with that of the ordinary rubber cement mortar. The mechanical and low-temperature tribology behaviors of PDA-rubberized mortars indicate a promising way to improve the service performance of the rubberized mortars and concretes.

Keywords Mussel inspired modification · Rubberized mortar · Interfacial hydrophilicity · Tribology test · Low temperature

1 Introduction

Stockpiling of thousands of waste rubber tires is a crucial environmental risk due to the extensive potential issues including water, air, and soil pollutions [1]. Recently, putting waste tires rubber crumbs into cement concretes attracted more research interest with

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the consideration of the usage of waste rubber tires [2, 3]. It is regarded as a resource-conserving and environment-friendly way to reuse solid waste. The intrinsic properties of crumb rubber, including the low stiffness, the outstanding flexibility, and the low apparent density, endow crumb rubber concrete the remarkable performances during the service life [4, 5]. It is demonstrated that various properties and performances of cement concretes can be upgraded with the addition of rubber crumbs. The effects of surface modifications of rubber crumb on the tribological properties of crumb rubber cement concretes have seldom been reported. Gonen et al. [6] investigated the effect of rubber crumbs on the mass loss due to the freeze–thaw cycles. It indicates that the mass loss of concrete reduces about 90% with 4 wt% addition of rubber crumbs. Thomas et al. [7] studied the invasion of chloride variation with rubber crumbs addition. The invasion length of the crumb rubber concrete is found reduced significantly compared with the plain concrete. Kumar et al. [8] demonstrated the obvious improvements of the abrasive resistance of the concrete with rubber crumbs addition. Wang et al. [9] claimed the anti-permeability of the foam concrete can be remarkably improved by using rubber crumbs. Although various benefits of crumb rubber concrete have been widely reported, the obvious reduction of the mechanical properties is an inevitable flaw [10–14] due to the weak interfacial adhesion. Inspired by the related studies [15], several efforts have been made to enhance the interfacial adhesions between rubber crumbs and the cement matrix. Surface modification technologies have been proposed to change the physico-chemical properties of rubber crumbs, including surface grafting functional molecules, cementitious materials pre-impregnation, addition of silica fume, ultraviolet radiation [16–20]. Mussel inspired sticky coatings, especially polydopamine (PDA) coatings are demonstrated to own the enormous adhesion ability to the catechol motifs [15, 21–23].

In this study, PDA is utilized to improve the surface hydrophilicity of rubber crumbs. Effects of the surface modification of rubber crumbs on the mechanical behaviors of the cement mortars are systematically studied. The tribological properties of the plain mortars and rubber cement mortars with different rubber crumb contents are investigated to establish the relationships between interfacial interactions and tribological behaviors. For comparable study, other polyphenols, including tannin acid and dihydroxyphenylalanine, are used to coat on rubber crumbs. Furthermore, the tribological behaviors have been carried out to exploring the effect of the interfacial polyphenols modification on wear resistance of rubber cement mortar at low temperatures. Thus, the results of the study provide a new thought for rubber concrete pavement in severe cold areas by considering the surface modification of crumb rubbers.

2 Materials and Methods

2.1 Materials

The waste tire rubber crumbs (60 mesh, with an apparent density of 1.16 g/cm^3) used in this study are purchased from Gold Molar Environmental New Materials Co. Ltd, China. Portland cement (PO. 42.5) with a specific gravity of 3.1 and the Chinese standard sand (See its composition in Table S1) are used in this research. Hydroxide (NaOH), potassium permanganate ($KMnO_4$), dopamine hydrochloride (DA), tannic acid

(TA), dihydroxyphenylalanine (DOPA) were purchased from Chengdu Kelong Chemical Reagent Co. Ltd, China. All the chemicals were used as received without further purification.

2.2 Surface Modification of Crumb Rubber

The raw crumb rubber (OR) were treated by 5 wt% NaOH solution for 24 h to remove the surface oil and then washed to be neutral. The NaOH treated crumb rubber were oxidized at 60 ℃ by 5 wt% $KMnO_4$ solution for 2 h at pH 2 to obtain the oxidized crumb rubber (OXR). Then the OXR were washed to be neutral again. The polydopamine (PDA), TA, and dopa modified rubber crumbs were prepared by treating the oxidized rubber crumbs with their solution for 12 h, respectively. The schematic diagram of modification is shown in Fig. 1a. After washing, the PDA-, TA-, and Dopa-modified crumb rubbers were used for the characterization. Fourier Transform Infrared Spectroscopy (FTIR) testing was used to identify the surface functional group variation on different modification crumb rubbers by a Fourier Infrared Spectrometer (Bruker Tensor—II, Germany). Samples (3–5 mg) are grounded together with a certain amount of KBr and are pressed into a thin sheet for testing. Then the dynamic and static contact angle characterizations were carried out to determine hydrophilicity variation of the pristine and the varied modified crumb rubbers.

2.3 Mortar Mixture Proportion

Five different types of rubber cement mortars were prepared to investigate the influence of crumb rubber surface modification on the interfacial behavior of rubber and hydration product of cement in mortar. These mortars involve of different types of crumb rubber, including the OR crumbs, OXR crumbs by NaOH and $KMnO_4$, PDA modified-, Dopa modified- and TA modified rubber crumbs. All the rubber crumb mortars were mixed with the sand: cement: gravel ratio of 3:1, water-cement ratio of 0.5. Four sets of mortar mixtures incorporating fine crumb rubber as 5%, 10%, 15% and 20% volume replacement of the standard sand were prepared for each type of rubber cement mortar. The mass variation of mortar with the addition of rubber is shown in Table S2.

2.4 Compressive Strength

Cubic mortar specimens with dimension 40 mm × 40 mm × 160 mm were prepared and cured in the standard curing conditions for 28 days before the compressive strength testing, in accordance with GB/T 17,671–1999. Then the universals testing machine was used to record the compressive strength of specimens (see Fig. S2a), each testing contains three parallel simples.

2.5 Tribology Evaluation

The tribological tests were performed using a pin/disc (PD) type (GDZ-1, China) wear tester under dry conditions, i.e., in air ambience without dealing with humidity issue with relative humidity ≈ 70% in a room. The schematic diagram of PD tests was shown in Fig. 1b. The friction pair is made of polyoxymethylene (POM) with the hardness close to that of automobile tires. Each test lasted 10 min with a speed of 7.95 rpm and the load of 50 N. The effect of load is tested by varying load from 10 to 100 N with an interval of 10 N. 40 N to 60 N is found a proper

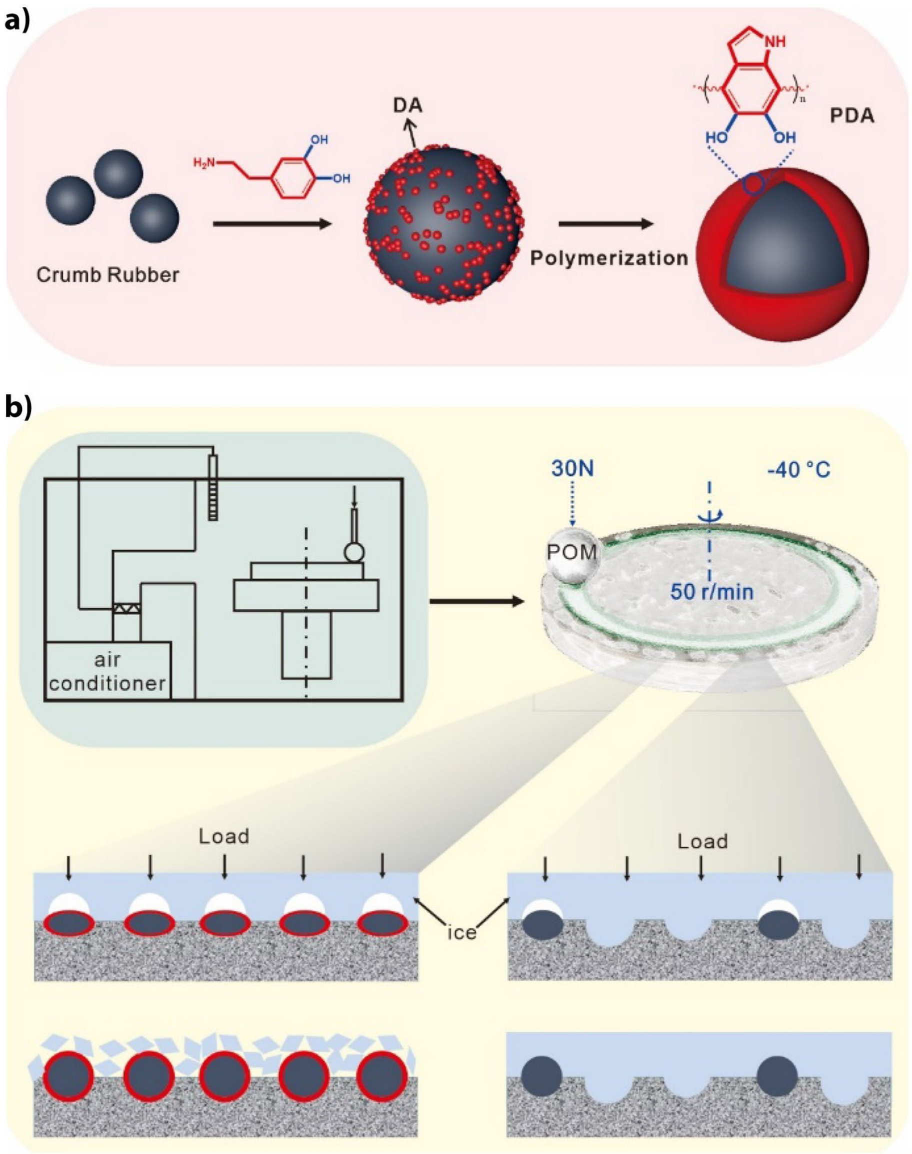


Fig. 1 (**a**) PDA modification of crumb rubber and (**b**) the schematic drawing of tribology test of rubberized mortars containing different types of crumb rubbers

load range for the wear testing by considering the weight loss and appearance of the samples. 50 N is chosen to mimic the speed and weight of the 30-ton truck with 12 tires. Cylindrical mortar specimens were casted with the size of Φ 3.4 cm×1.5 cm. Each test was carried out under 20℃, 0℃, -20℃ and -40℃ respectively with the similar procedure of the reports [24, 25]. Temperature control is achieved by continuously spraying liquid nitrogen on to the friction pair. The samples used for the test are shown in Fig. S2b. The testing procedure refers to the GB/T 16,925–1997. A fish bone diagram is used to show the research thoughts in Fig. S1.

3 Results and Discussion

3.1 Polyphenols Modification of Rubber Crumbs

Due to the apparent differences between the mass of the crumb rubber and the inorganic components including the cement and the sand, crumb rubber trends to float up and resulting in the separation between the inorganic and organic phases. Among the several methods to improve the dispersion of crumb rubber in mortar, surface modification exhibits the attractive effects for the dispersion of crumb rubber in mortar depending on its surface hydrophilicity largely. Here, three different polyphenols including PDA, TA and DOPA are applied to enhance the surface hydrophilicity of crumb rubber and comparing with the pristine or the OXR modified crumb rubbers. Figure 2 shows SEM images, EDX mapping images and FTIR spectra of the different types of crumb rubbers to illustrate their hydrophilic differences. The island like pristine crumb rubber changes to the relative dense particle coated morphology after OXR modification, as shown in Fig. 2a, c. Interestingly, the microstructure of crumb rubber is more uniform and denser after PDA modification, as shown in Fig. 2e. Meanwhile, the EDX results also indicate that the PDA modification can improve the surface oxygen element contents on crumb rubbers obviously, as shown in Fig. 2b, d, f. The peak area of 3600 cm^{-1} increase after PDA modification comparing to that of the pristine crumb rubber which indicates the substantial increase of the hydroxyl group content. Thus, the PDA-modified crumb rubber should be hydrophilic based on a large number of surface hydroxyl groups. The surface morphology variation of rubber crumbs before and after modification by TA and DOPA is shown in Figs. S3 and S4. The increasing of hydroxyl groups content was also found. Figure 3 shows the results of the static and dynamic water contact angle testing and the fluidity of crumb rubber-contained cement mortar. It can be seen that the water contact angle of crumb rubber decreases from 149° to 114° after OXR modification, and it decrease to about 84° with the PDA modification. Polyphenol modification rubber crumbs show improved hydrophilicity, compared with NaOH, sulfonation and urea modification reported in the literature [26]. To explore the stability of the hydrophilicity of PDA-crumb rubber, the dynamic water contact angle of different types of crumb rubber is studied according to the Washburn theory. The increase in liquid volume into the pores of rubber crumb can be used to reflect the dynamic hydrophilicity. The Washburn theory is described in the following equations:

$$T = \left(\frac{\eta}{C\rho^2\gamma\cos\theta}\right)M^2 \tag{1}$$

$$\cos\theta = \alpha \times \frac{M^2}{T} \tag{2}$$

where T refers to the contact time, η is the viscosity of water, C refers to material constant characteristic of crumb rubber, ρ refers to the density of water, γ refers to the surface tension of water, θ refers to the contact angle, and M stands for the mass of water absorbed on crumb rubber. According to Fig. 3b, the mass of water adsorbed on polyphenols modified crumb rubber increased apparently comparing those of the OXR-modified and the pristine crumb rubbers. This indicates that the dynamic water contact angle of crumb rubber by polyphenols modification can effectively enhance. Moreover, PDA modification is the most effective one among the three. The effect of the surface hydrophilic modification of crumb rubber on the workability of the crumb rubber-cement mortar was also analyzed by

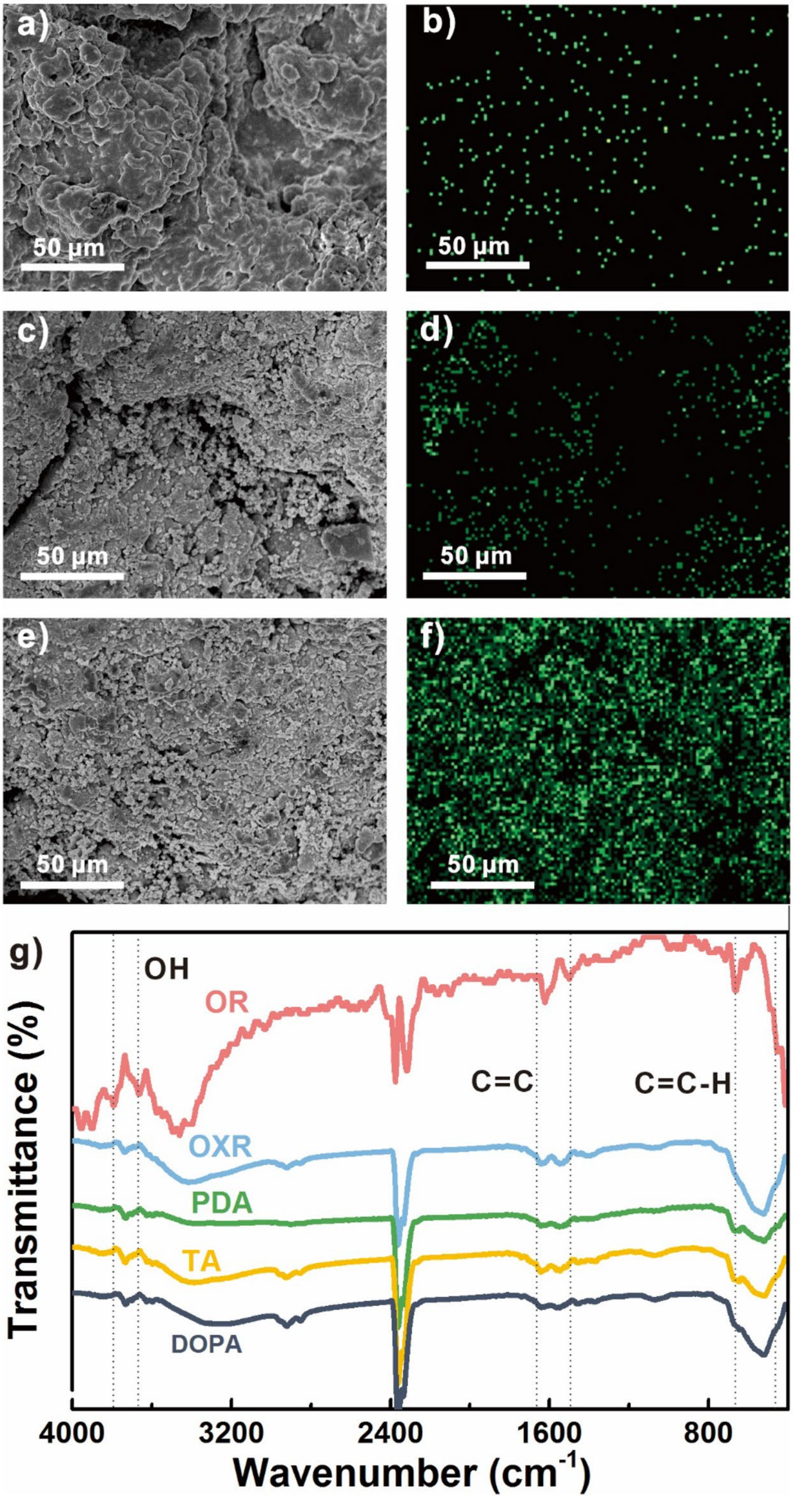
Fig. 2 (**a**), (**c**), and (**e**) SEM images of the OR-, OXR- and PDA-modified rubber, (**b**), (**d**), and (**f**) The EDX mapping of the OR-, OXR- and PDA-rubber for oxygen element (green colors), and (**g**) The FT-IR spectra of the pristine and the surface-modified crumb rubbers ▸

the fluidity characterization. As it is shown in Fig. 3c, the PDA modified crumb rubber-cement mortar exhibits an obvious improvement in fluidity than that of the pristine crumb rubber-cement mortar. Thus, based on the above analysis, polyphenol modification can be an effective way to improve the hydrophilicity of crumb rubber significantly.

3.2 Effect of Surface Modification on Compressive Strength of Rubber Cement Mortars

The effect of the different surface modifications on the compressive strength of rubber cement mortars is systematically studied. Figure 4 shows the compressive strength variation of rubber cement mortars at different curing times. Meanwhile, the effect of the rubber content on the compressive strength of rubber cement mortars is also explored. It indicates that the polyphenols modification of rubber reduces the compressive strength of rubber-cement mortars comparing with that of the after 3-d curing. Comparing with OR, the compressive strength of OXR-, PDA-, TA- and DOPA-modified mortars decreased by 25%, 32%, 29% and 34% respectively. An apparent change of compressive strength can be observed with the increase of the curing time, polyphenols-modified rubber-cement mortars increased significantly which is higher than that of OR. The OXR-, PDA-, TA- and DOPA-modification can improve the compressive strength of rubber cement mortar by 17%, 37%, 20% and 33% after 28-days curing, respectively. Table 1 compares the results of this work with published literatures to show the effect of polyphenol modified rubber crumbs. Besides, the compressive strength of rubber-cement mortars decreased with the increase of the rubber contents, as shown in Fig. 4b and evidenced by others [27–29]. The variation of the compressive strength is attributed to the phase variation in the interface transition zone (ITZ). Comparing the phase composition of the pristine rubber with that of the PDA modified rubber, the Ca/Si ratio decrease from 4 to 2.2 (see Table 2). This indicates more hydrated calcium silicate formed [30, 31] in the ITZ of the PDA modified rubber mortar.

An apparent gap can be seen in the ITZ of the pristine rubber-cement mortar, while for the PDA and TA modified rubber-cement mortars, the closely contacting interface between the modified rubber and cement matrix in the ITZ can be intuitively found. This is ascribed to the hydrophilicity variation of the different rubbers. Water trends to aggregate closing to the hydrophilic surface of the modified rubber, thus the hydration of cement is delayed in the ITZ closing to the rubber in polyphenols modified crumb rubber-cement mortar (Fig. 4). The compressive strength of polyphenols modified rubber- cement mortar is lower than that of OR–mortar at the early hardening in turn. With the hardening of cement, water-saturated around the modified crumb rubber gradually, which is beneficial for the cement hydration and improving the content of hydrated calcium silicate. Thus, the organic–inorganic interfacial adhesion is significantly enhanced by the polyphenol modification.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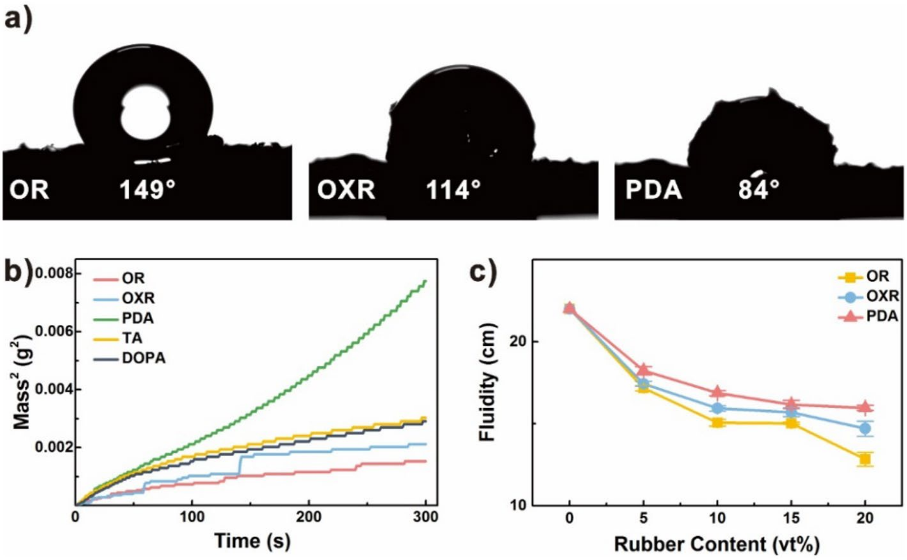

Fig. 3 (**a**) Static contact angle testing situations before and after modification of crumb rubbers, (**b**) Dynamic contact angle testing situations before and after modification of crumb rubber, and (**c**) Influence of crumb rubber before and after modification on the fluidity of cement mortar

3.3 Abrasion Resistance of the Rubber Cement Mortars

The outstanding mechanical deformability and frost resistance of rubberized concrete attracts many research interests aiming to apply the materials in the cold regions'

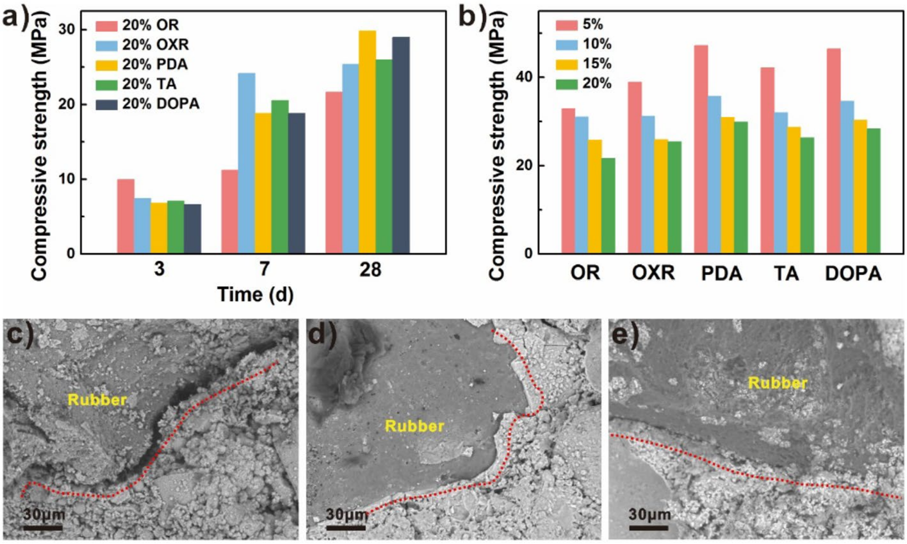

Fig. 4 Compressive strength of (**a**) different crumb rubber-cement mortars at different curing time, (**b**) the 28-d compressive strength of different crumb rubber-cement mortars with different rubber content. SEM images of cross sections of the (**c**) OR-, (**d**) OXR- and (**e**) PDA-mortars after compressive strength test

Table 1 Improvement of compressive strength of rubberized concrete by modification method

Rubber powder modification method	Rubber content (Vol %)	Compressive strength (MPa)	
		Control	Mixing with rubber
PVA [1]	20	45.0	30.0
$KMnO_4$ for 2 h, then $NaHSO_3$ for 1 h [17]	4	49.2	23.6
NaOH for 24 h [34]	20	55.6	27
Water soaking for 24 h [35]	20	35.4	21.5
Cement and silane precoating [36]	30	38.0	23.0
Cement paste pre-coating [37]	38	54.0	32.0
This study	20	43.7	29.8

infrastructures. A high enough friction coefficient between transportation and concrete pavement should be sustained to guarantee the infrastructure and traffic safety. In cold regions, the thin ice layer formed rubberized concrete pavement affecting their tribology to a great extent. Here, tribology test is carried out to explore the influence of polyphenols modifications of crumb rubber on the abrasion resistance of mortar. Figure 5 shows the coefficient of friction (μ) evolution over the temperature and rubber content for the different crumb rubber-cement mortars. It indicates that the abrasion resistance ability of mortar improved with a certain content of crumb rubber addition. The abrasion resistance ability of mortar decreased when the crumb rubber content is more than 5% (volume ratio). The phenomenon can be found in four different testing temperatures, including 20 ℃, 0 ℃, -20 ℃ and -40 ℃. Among the four modified crumb rubbers, i.e., OXR-, PDA-, TA-, DOPA-modified, PDA-modified crumb rubber plays a relatively interesting role in the abrasion resistance ability improvement for mortars. Although the μ of PDA-modified rubber-cement mortar is larger than that of the ordinary cement mortar (OC) under 20 ℃ with 5% rubber content (volume fraction). The μ of PDA-modified rubber—cement mortar is about 0.82, which is smaller than that of the other four mortars (0.97, 0.94, 0.90, and 0.89 respectively for the OR- cement, OXR-, TA-, and DOPA- modified rubber—cement mortars). With the decreasing of temperature, the decreased amplitude of μ of PDA-modified rubber—cement mortar is the smallest one among these rubber-cement mortars. The μ of PDA-modified rubber—cement mortar is 0.64 under -40 ℃, which is close to that of OC under 0 ℃. In addition, the wear value of rubber cement mortar is reduced by polyphenol modification with Table 3. It indicates that the PDA-modified rubber-cement mortar exhibits outstanding abrasion resistance under the low-temperature condition.

To reveal the influence of polyphenols modifications on the tribology properties of rubber-cement mortar and further elucidate the tribological mechanism, the micro-morphology of six mortars surfaces after the tribology test are analyzed (Fig. 6). Apparent abrasive dusts of friction pair (ball) adhere onto the OC mortar surface indicates its excellent abrasion resistance due to the relatively high hardness. While with the addition of

Table 2 The Ca/Si ratio of rubberized cement mortar

Rubberized cement mortar	Ca (wt%)	Si (wt%)
OR-	41.17	9.48
PDA-	32.12	14.41

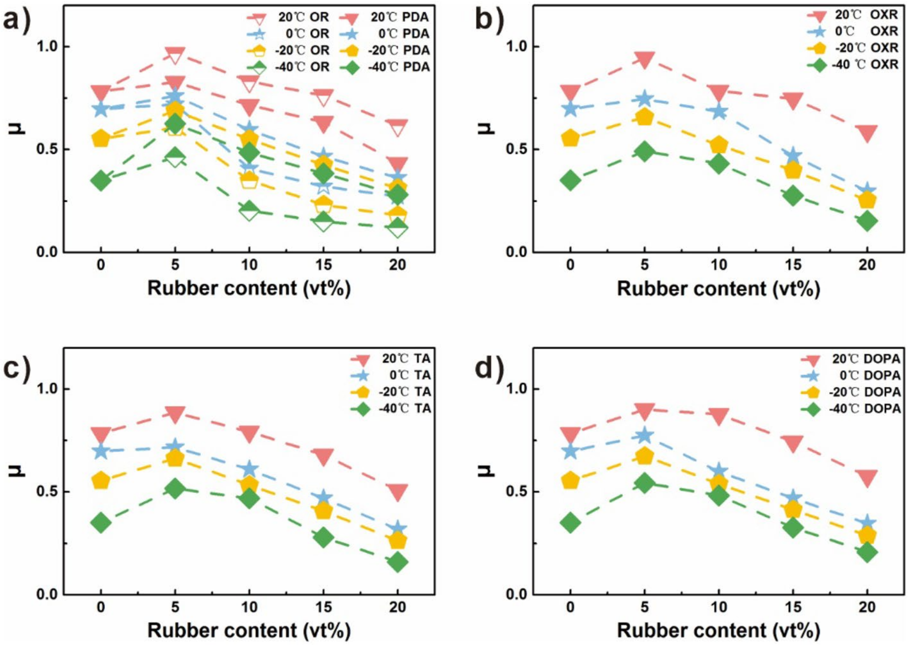

Fig. 5 The coefficient of friction (*μ*) variation over the temperature and rubber content for the different crumb rubber-cement mortars (**a**) PDA-, (**b**) OXR-, (**c**) TA- and (**d**) DOPA-

rubber, the abrasion resistance properties of mortars are increased due to the flexibility of crumb rubbers. Nevertheless, without surface modification, crumb rubber can easily fall-off from the mortar surface and the friction pair slide on cement matrix directly at the local area. While after surface modification, with the enhanced interfacial adhesion between crumb rubber and cement matrix, the fall-off phenomenon for crumb rubber cannot be found. Thus, although the μ of OR-cement mortar is the highest one among all rubber mortars, it is not the real μ due to the damage of the specimen (Fig. 6b). According to the analysis above, the polyphenols modification of rubber, especially PDA-modification can significantly improve the abrasion resistance of mortar at low temperatures. As indicated by the results of SEM and EDX (See Fig. 2), the shedding is severe for the rubberized mortar without surface modification. While PDA modification can obviously reduce the shedding amount of the mortar after wear. Besides, according to Table 3, it is also found that PDA and other modifications could improve the wear resistance of rubberized mortar.

Table 3 Weight loss of different mortars after tribological testing at -40℃

Crumb rubber cement mortars	Mass before friction (g)	Mass after friction (g)	Weight loss(g)
OR	65.37	61.59	3.78
OXR	66.81	64.20	2.61
PDA	62.67	61.75	0.92
TA	63.59	62.23	1.36
DOPA	65.77	64.70	1.07

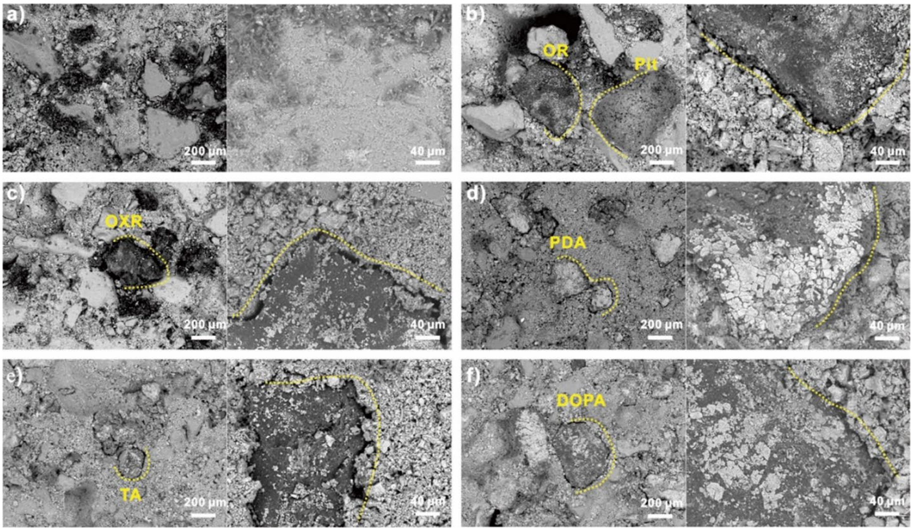

Fig. 6 SEM images of the different crumb rubber -cement mortars after wear test (**a**) OC-; (**b**) OR-; (**c**) OXR-; (**d**) PDA-; (**e**) TA- and (**f**) DOPA-mortar

Thus, for the ordinary rubberized mortar, the rubber crumbs are easy to fall from matrix during wear process, and the adhesion of ice on the surface is relative strong. While, for the PDA modified rubberized mortar, it is hard for rubber crumb to fall from matrix during the wear process, which is also demonstrated by the variation of the wear content of the different samples (See Fig. S5). The ice layer is easier to breaking and falling due to the apparent stress difference between the soft rubber crumbs and the hard cementitious materials, and resulting in the higher friction coefficient.

3.4 Mechanisms of Polyphenols Modified Mortars' Tribology at Low Temperature

The various friction performance of cement mortar is mainly due to the different roughness caused by surface microstructure. The incorporation of rubber increases the roughness of cement mortar, and an increase can be observed in the friction coefficient. However, the adsorbed water forms an ice condensation layer on the cement mortar in a low-temperature environment to reduce the surface roughness. The previous study has shown that the condensation layer will act as a lubricant and reduce the surface's friction coefficient [32]. Interestingly, an interface layer with uneven hardness can be formed with the addition of crumb rubber, and the deform occurs with the loading of stress, which eventually leads to the generation of ice layer' crack and increases surface roughness. It is similar with the ice breaking mechanism of the pavement filled by the polyurethane elastomer [33]. Among them, the combination of crumb rubber and cement mortar is a key parameter that determines its friction performance. In the pure rubber cement mortar, the poor rubber concrete bonding force causes the rubber to fall off. The lack of deformable crumb rubber makes it difficult to destroy the ice layer and builds a rough surface to improve the friction performance. Fortunately, mussel-inspired polyphenol modification provides new strategies to enhance the interfacial adhesion of crumb rubber and cement mortar. Due to the modification of polyphenols, the abundant catechol functional groups on the rubber powder's surface can effectively improve rubber and cement mortar wettability, provide strong interfacial bonding ability, and make its long-term storage in cement mortar test blocks stable (See Fig. S6). The long-term existence of rubber powder can maintain the cement mortar surface's long-term ice-breaking ability and enhance the surface's low-temperature wear resistance.

4 Conclusions

In this study, mussel inspired polyphenol modified crumb rubber has been added in cement matrix and tribology properties of rubber-contained cement mortars has been characterized with the promising application in cold regions.

1. Mussel-inspired polyphenol modification improves crumb rubbers' surface hydrophilicity by introducing polar functional groups. The modified crumb rubber shows strong interfacial adhesions with cement, which exhibits an outstanding surface tribology property at low temperatures.
2. The hydration behavior of cement around hydrophilic crumb rubbers is regulated by the variation of the surface hydrophilicity. Water molecules and Ca^{2+} tend to aggregate around the hydrophilic crumb rubbers surfaces and result in the retarding of the cement by decreasing the water content contacting with cement. The compressive strength of rubberized mortar modified by PDA, TA, and DOPA decreased by 32%, 29%, and 34% respectively after 3 days of curing. These aggregated water molecules guarantee the enough supply for the cement hydration and strengthen the rubberized concrete later. The compressive strength of rubberized mortar modified by PDA, TA, and DOPA increased by 37%, 20%, and 33% respectively after 28 days of curing.
3. The friction coefficient of cement mortars is gradually decreased as the temperature decrease. The addition of modified crumb rubber can apparently delay friction coefficient reduction. Among the various modified rubberized mortar, the friction coefficient of PDA modified rubber cement mortar decreased by only 28%.
4. Favorable friction performance of polyphenol modified rubberized mortars at low temperatures enables the potential applications in pavement constructions in cold regions.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10443-021-09938-3.

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Data Availability Statements Raw data of tribological testing were generated at the pin/disc (PD) type wear tester (GDZ-1, China). Derived data supporting the findings of this study are available from the corresponding author upon request.

Declarations

Conflict of Interest The authors declare no competing financial interest.

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