

Article

Timely and Durable Polymer Modified Patching Materials for Pothole Repairs in Low Temperature and Wet Conditions

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Abstract: Aiming to solve the contradiction between the workability and performance of pothole patching using cold mixtures, this paper proposed new patching materials based on the microcapsule technique and polymer reinforcement, namely cold mixtures with polymer modified asphalt and dense graded (DG) of aggregates (PADG) mixtures. Laboratory tests were conducted to compare the PADG mixtures with commonly used DG mixtures and open graded (OG) mixtures concerning workability, storability, cohesion, stability, and durability of each mixture. The results found that the PADG mixtures were satisfactory in their workability and storability and were as satisfactory as the OG mixtures. Meanwhile, stability and durability of the PADG mixtures was better than the DG mixtures and OG mixtures, i.e., the PADG mixtures showed stability in the conditions of the timeliness, low temperature, and immersion, as well as the freeze-thaw resistance and wear resistance in wet conditions. Therefore, it can be concluded that PADG mixtures are applicable in timely and durable pothole repairs in low temperatures and wet conditions.

Keywords: pothole repairs; patching materials; microcapsule; polymer; low temperature; wet

1. Introduction

In general, pothole problems can be defined as bowl-shaped holes in the surface of asphalt pavement, and their existence is averse to traffic safety and rideability of roads [1,2]. Potholes often develop from heavy traffic loads in adverse weather, especially in winter or rainy seasons [3,4]. Water is the critical factor in aggravating the distress of potholes, which can seep into the internal pavement through low-temperature cracking due to deformation or alligator cracking due to fatigue [5,6]. Finally, the water damage leads to structural failure and the hollowing out of the base or subgrade. Furthermore, low temperatures and water have negative effects on the patching process and durability of pothole repairs [7,8]. Thus, timely and durable repairs are essential in order to prevent further deterioration.

The most common distresses of pothole patches include shoving, raveling, dishing, bleeding, edge disintegration, stripping, and alligator cracking. These distresses are closely related to the quality of binder and the rationality of gradation [5,8,9]. According to material composition and construction technology, patching materials can be divided into three groups: hot-mixed and hot-placed patching mixtures; hot-mixed and cold-placed patching mixtures; and cold-mixed and cold-placed patching mixtures [10–13]. The last two using cutback asphalt and emulsified asphalt as their binder are preferred in winter, as they do not require heating [14–16]. However, these two kinds of patching mixtures have rigid requirements for the temperature during construction [11]. There are continuous

gradation and gap-graded gradations in cold mixtures, and continuous gradation has a superior pavement performance but has poorer workability, as compared with gap graded gradation. [3]. Thus, the selection of the binder and gradation has a significant impact to the design of patching materials.

The cold mixtures solidify gradually with the increase of storage time due to the volatilization of the dilute and curing of the additive in the binder, which has an adverse effect on the workability of the cold mixtures during the construction process. Ideally, microcapsule technology can effectively solve this problem. A microcapsule is a type of micro-container, with an outer shell wall made of natural or synthetic polymer material [14,17,18]. It effectively inhibits the reaction of the active material with the external environmental factors and reduces the diffusion and evaporation of core materials into the environment [19,20]. In recent studies, Choi et al. controlled the cement hydration reaction by encapsulating the additive in an effort to reduce the cracking caused by hydration heat of concrete, during extreme weather [21]. Perez et al. developed the self-repairing concrete by using epoxy-containing silica microcapsules and amine-functionalized nanosilica [22]. Then, Wei Du et al. found that microcapsules with toluene diisocyanate (TDI) used as the core and paraffin used as the shell, could be prepared through the melting condensation method allowing for self-healing concrete. The compressive strength of mortar can be increased by 28.2% [23]. Daquan Sun et al. developed an aided regeneration system of aged asphalt binder based on the microcapsule technology, which can prolong the service life of the asphalt pavement. Microcapsules containing regenerants were prepared by in-situ polymerization. The capsule shell was made of melamine-urea-formaldehyde (MUF) resin [24].

The main objective of this paper is to propose new polymer modified using dense graded patching materials (i.e., polymer modified asphalt and dense graded (DG) of aggregates (PADG) mixtures). This is based on microcapsule technique and polymer reinforcement, and verifies the practicability of PADG mixtures allowing for timely and durable pothole repairs in winter-rainy conditions. Past studies have summarized the main properties of cold mixtures, such as workability, storability, stability, stripping resistance, freeze-thaw resistance, durability, bonding performance and skid resistance [1,8,9]. Laboratory tests were conducted to evaluate the pavement performance of the PADG mixtures, compared with the two typical patching materials of DG cold mixtures (i.e., DG mixtures) and open graded (OG) cold mixtures (i.e., OG mixtures), including workability, cohesion, storability, bonding performance in low temperature and wet conditions, and durability.

2. Materials and Methods

2.1. Materials

As shown in the Figure 1, the PADG mixtures consisted of 70.1% aggregate, 23.4% mineral fines, 5.5% matrix asphalt, and 1% additive. Among these, the functional components of the additive involved the modifier, adhesive, and a film-forming agent. Thirteen percent Styrene–Butadiene–Styrene (SBS) polymers was used as a modifier and were added to 18% of trichloroethylene evenly, which was from Shandong Agile Road Bridge Engineering Co., Ltd., China. The adhesive was made up of 15% ethylene-vinyl acetate copolymer (EVA), which was supplied by Zhejiang Minghua new material co., Ltd., China, and it was dissolved into 34% 2-Ethoxyethyl Acetate. 10% polystyrene was used as the film-forming agent. The plasticizer was 10% di-n-butyl phthalate, which was provided from Nanjian Chemical Co., Ltd. The chemical formulas for the active component in additive are shown in Figure 2.

The Fourier infrared spectra of the matrix asphalt and the new polymer modified asphalt are shown in Figure 3. The peak of 1594 cm^{-1} represents a conjugated olefinic bond from the film-forming agent, which was used to form a flexible solid film within the spatial network structure of the microcapsules. The microcapsules containing plasticizer were spread into the additive evenly, as shown in Figure 4. This was done to ensure the workability and storability of the cold mixtures by isolating the modifier or adhesive and plasticizer in the microcapsules during the storage period. When a constant and sufficient pressure is loaded on the capsules, the capsules rupture and the plasticizer flow out of the

capsules to catalyze the curing of modifiers and adhesive. In order to guarantee the stability of the microcapsules during the storage phase and the breakability of the microcapsules in the use stage, Microcapsules require a specific toughness and density. Therefore, the values of n_6 in Figure 2c are recommended which are between 800 and 1200. The peak in wavenumber 965 cm^{-1} was functional groups of polybutadiene from the modifier, which can improve the low temperature performance of the binder. The absorption peaks in the range of $2800\text{--}3000\text{ cm}^{-1}$ were caused by the C-H stretching vibration of cycloalkanes and alkanes, which is one of most common chemical bond in asphalt [25,26].

90# matrix asphalt is produced in Korea, and its main properties were listed in Table 1. Compared with matrix asphalt, the cured polymer modified asphalt has a higher softening point of $55.8\text{ }^\circ\text{C}$. Both aggregates and fines are made of limestone. The OG mixtures and the DG mixtures, produced in Canada and China, and are widely used as patching materials in pothole repairs. The gradations of mixtures are plotted in Figure 5.

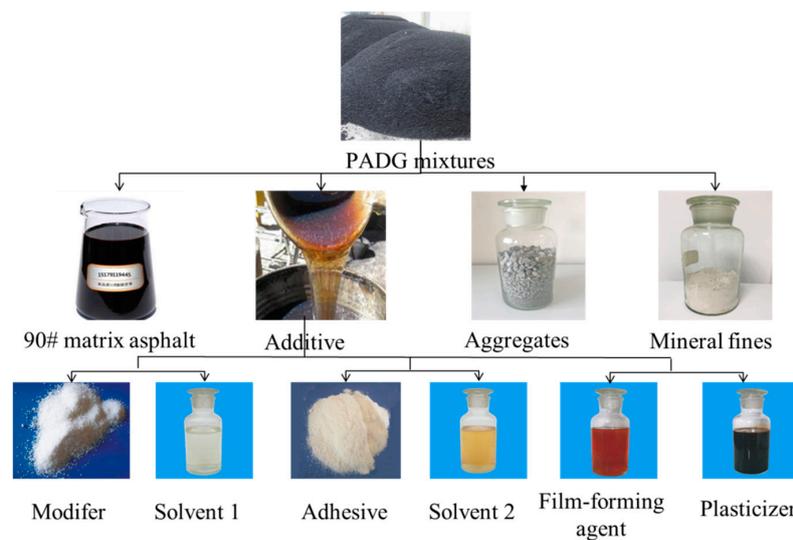
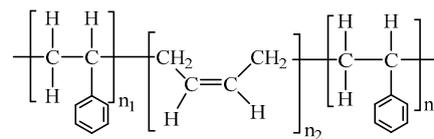
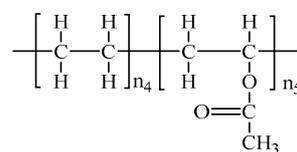


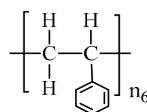
Figure 1. Compositions of polymer modified asphalt and dense graded (DG) of aggregates (PADG) mixtures.



(a)



(b)



(c)

Figure 2. The chemical formulas for the active component in additive: (a) Styrene–Butadiene–Styrene (SBS) polymers; (b) ethylene–vinyl acetate copolymer; (c) polystyrene.

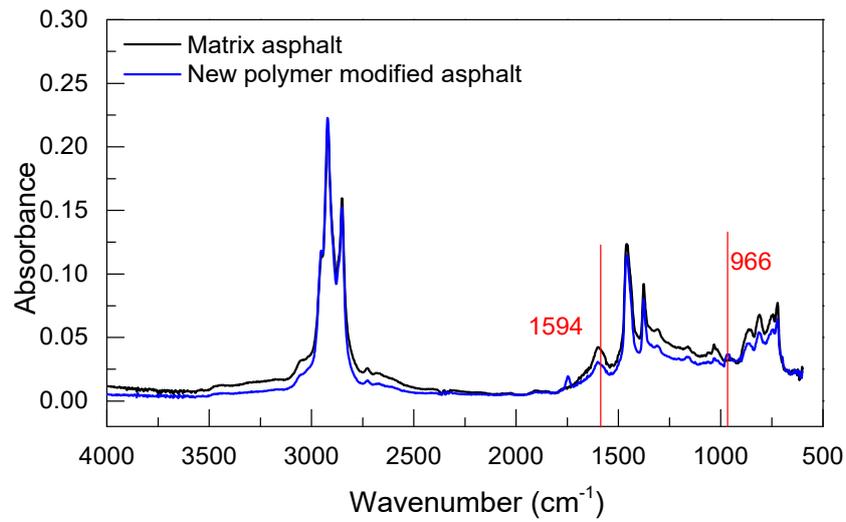


Figure 3. Fourier infrared spectra of matrix asphalt and new polymer modified asphalt.

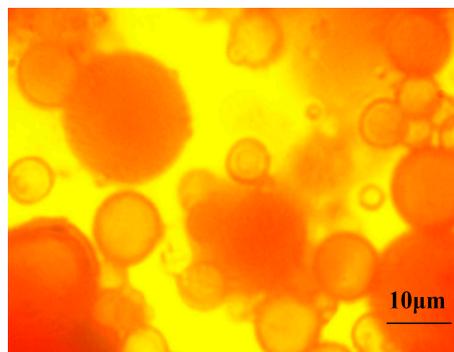


Figure 4. Microscopy images of microcapsules in additive.

Table 1. The main properties of 90# matrix asphalt.

Properties	Values	Specifications
Penetration (25 °C, 100 g, 5 s) (0.1 mm)	88.5	80~100
Softening point (°C)	48.2	≥44
Ductility (15 °C) (mm)	138	≥100
Density (g/cm ³)	1.022	-
Wax content (%)	1.6	≤2.2

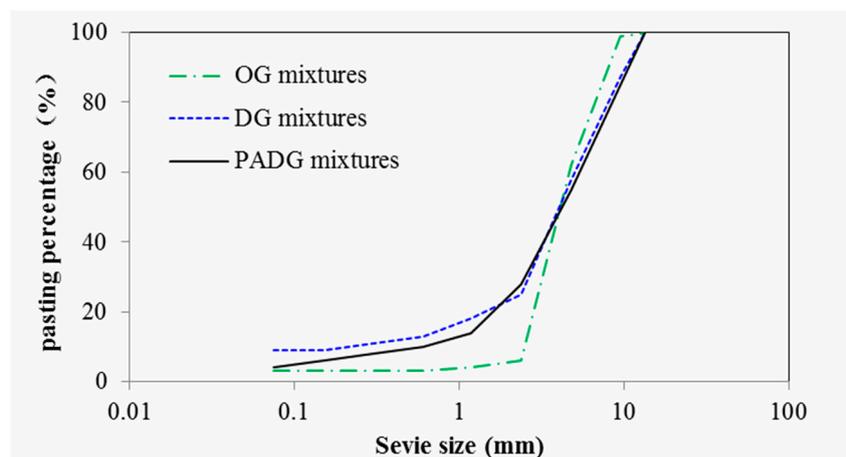


Figure 5. Gradations of DG mixtures, open graded (OG) mixtures and PADG mixtures.

2.2. Methods

2.2.1. Workability and Storability Tests

The workability of the cold asphalt mixtures refers to the patching materials retaining a proper loose state to shovel, dig, screed and compact easily during the installation process. Today, the workability test methods include the PTI/SHRP Workability Test (AASHTO TP43-94), the Pennsylvania Department of Transportation (PennDOT) spatula method, and the opinions of the maintenance workers [8]. However, the test methods mentioned above are influenced by the subjectivity of the experimenter, and there are differences between the test methods and the workability of the mixtures in the construction process. Therefore, in this study, the workability and storability test method have been improved. Three thousand grams of PADG mixtures, DG mixtures, and OG mixtures were stored for 48 h under 100 kg loads at 5 and 20 °C, respectively. As shown in Figure 6, a working stirring tester was used to measure the torque when patched different types of cold mixtures. The average torque of the first minute represents the magnitude of the paving friction. The greater paving friction characterizes the worse workability of mixtures. Storability of the patching materials was evaluated by testing the workability of cold mixtures, which were packed into sealed bags at 5 °C under 50 kg load for 3, 7, 30, 60, 120, and 240 days. Three parallel tests were conducted in per test condition.

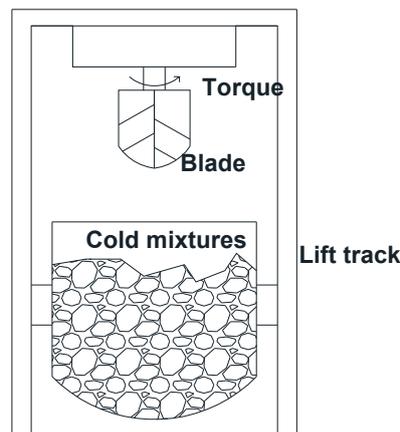


Figure 6. Principle of working string tester.

2.2.2. Cohesion Tests

Patching materials with poor cohesion will induce distress in winter or rainy weather conditions, such as raveling and debonding. Therefore, conducting a cohesion test is an essential measurement in cold mixture design. In this study, the cohesion test procedures were conducted according to the report of FHWA-RD-99-168 [2]. Eight hundred grams of PADG mixtures, DG mixtures and OG mixtures were put into the standard Marshall mold and placed into a consistent-temperature room at 4 °C for 24 h. Then, using a standard Marshall hammer (4.5 kg), five blows were loaded onto the both sides of the Marshall sample. The Marshall sample was put onto a standard sieve with 25.4 mm openings and rolled back and forth 20 times along the upright sieve frame. The sieve was laid and sample pieces fell from the meshes for 10 s. The cohesion of the mixtures is characterized by the percentage of materials retained on the sieve. The porosity of the PADG mixtures and the DG mixtures were about 5.5 and 3.8%, respectively, while the OG mixtures had a larger porosity of 12.0%. Three parallel tests were conducted in per test condition. Hence, a total of nine Marshall specimens were prepared in cohesion tests.

2.2.3. Marshall Tests

Marshall tests were used to evaluate the stability of the PADG mixtures, DG mixtures, and OG mixtures. The Marshall tests followed the procedure of ASTM D 1559 [27]. The contents of the air pores

in the Marshall samples were 5.5% (PADG mixtures), 3.8% (DG mixtures), and 12.0% (OG mixtures). Before completing the Marshall tests, the standard specimens were pre-treated to assess representative stability in adverse conditions in the following manner:

1. Initial stability: Three standard Marshall samples per mixture were stored in a ventilated room for 2 h at 25 °C.
2. Molded stability: Three standard Marshall samples per mixture together with mold were compacted 25 times again on both sides, after being side upright placed in an oven at 100 °C for 48 h. Subsequently, samples with mold were placed into a ventilated room of 25 °C for 48 h. Then the molds were removed and samples were placed in ventilated room for 2 h.
3. Stability forming speed: Three standard Marshall samples per mixture were placed in a 5 °C ventilated room for 3, 7, 15, 30, 45, and 90 days.
4. Low temperature stability: Three standard Marshall samples per mixture were placed in a refrigerator at −10 °C for 72 h.
5. Immersed stability: For this process there were multiple groups of samples. In the first group, three Marshall samples of per mixture were immersed in a 10 °C water bath for 30 min and then placed in 25 °C ventilated room for 72 h, to determine the dry stability (S1). As for the second group, the remaining three Marshall samples of each mixture were laid into a 25 °C ventilated room for 24 h after being soaked in a 10 °C water bath for 48 h. Marshall stability of immersed samples was tested and recorded as S2. The percentage rate of S1 and S2 was calculated to assess the ability of resistance to water damage.

2.2.4. Freeze-Thaw Tensile Strength Tests

Moisture damage, especially during winter, is considered one of the main distresses that cause the formation of potholes and require patching. That often causes or intensifies other types of distresses. In this paper, the Modified Lottman test AASHTO T-283 was conducted to determine the indirect tensile strength (ITS) of the PADG mixtures, the DG mixtures and the OG mixtures in a dry condition and after a freeze-thaw. The tensile strength ratio (TSR) of the ITS before and after freeze-thaw was calculated to evaluate the long-term water susceptibility of cold mixtures [28].

In this test, there were two subsets of Marshall samples. The first group included a dry subset which was stored at room temperature for 40 h. The second subset was pre-treated with freeze-thaw, the process was as follows. Samples were immersed into water immediately after vacuumed in order to control the saturation degree range from 70 to 80%. Then the vacuum-saturated specimens were tightly covered with a plastic film and placed into a refrigerator at a temperature of −18 °C for 16 h. Then specimens were removed and placed into a water bath at 10 °C for 24 h. The air pore content of the PADG mixtures, the DG mixtures, and the OG mixtures during the freeze-thaw tensile strength tests was the same to that in cohesion tests and Marshall tests. These three parallel tests were conducted in per test condition. Therefore, a total of 18 Marshall specimens were prepared in the cohesion tests.

2.2.5. Accelerated Loaded Test

The accelerated loaded test is a simulative test used to predict the dishing depth of the pothole patching caused by long-term vehicle load in moisture condition. Figure 7a describes the sample fabrication procedure. To begin with, two 30 × 30 × 5 cm AC-13 slab specimens were prepared. The next stage of pothole preparation process involved that two specimens were stuck together, after the middle section of the upper specimens was excised with the size of 30 × 13 × 5 cm. Next, coating materials were brushed on the surface of pothole evenly with the content of 1.0 kg/m². Subsequently, specimens were compacted 24 times with a 9 kN, and the specimens were patched using PADG mixtures, DG mixtures and OG mixtures. Finally, the composite plate was placed into an oven at 80 °C for 96 h and then taken out and placed in a ventilated room at an ambient temperature for 48 h. The objective of this process is to make sure the diluent in the cold mixtures volatilizes fully. The porosity of the PADG

mixture specimens, the DG mixture specimens and the OG mixture specimens were 5.2%, 4.0%, and 13.0%, separately. As shown in Figure 7b, a wheel pressure of 0.7 MPa was loaded on the composite plates at 30 °C, under immersed or dry conditions. The unidirectional rolling speed was 21 times/min. The dishing depth of pothole patching at middle of composite plates was measured after 2400 times wheel loads.

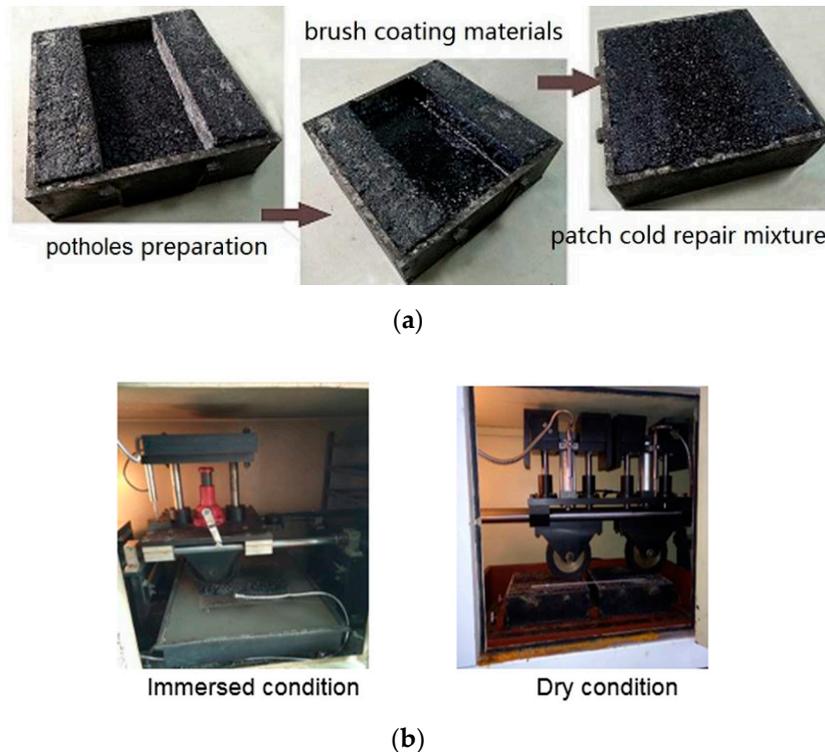


Figure 7. Accelerated loading tests: (a) The sample fabrication process of potholes patching durability tests; (b) accelerated loading tests device.

3. Results and Discussion

3.1. Workability Analysis

It can be seen in Figure 8 that the PADG mixtures and OG mixtures had a similar paving friction, which were both smaller than the paving friction of the DG mixtures, when at the same temperature. This indicates that the cold PADG mixtures and OG mixtures have a strong workability, due to the microcapsule technique and the gap-grading gradation separately, during the construction process. In addition, the level of the paving friction was also affected by the temperature. The higher the temperature, the better the workability of the asphalt binder.

3.2. Storability Analysis

It is particularly important that cold mixtures can be stockpiled with good workability for several months to patch potholes in time in adverse weather. As shown in Figure 9, the paving friction of the DG mixtures increased with the increase of storage time, but PADG mixtures and OG mixtures remained in a relatively stable state. The variation coefficient of the paving friction from the DG mixtures, when at different storage times was 4%, as compared with OG mixtures and PADG mixtures of 0.89% and 0.87% separately. Because the plasticizer was separated from the modifier and adhesive by microcapsules in PADG mixtures, that inhibited the curing of binder.

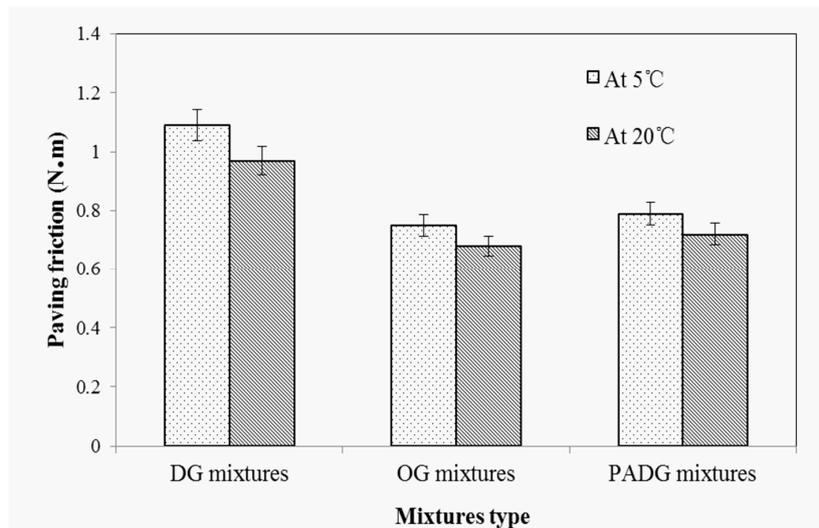


Figure 8. Paving friction of cold mixtures at different temperatures (at 5 °C and 20 °C).

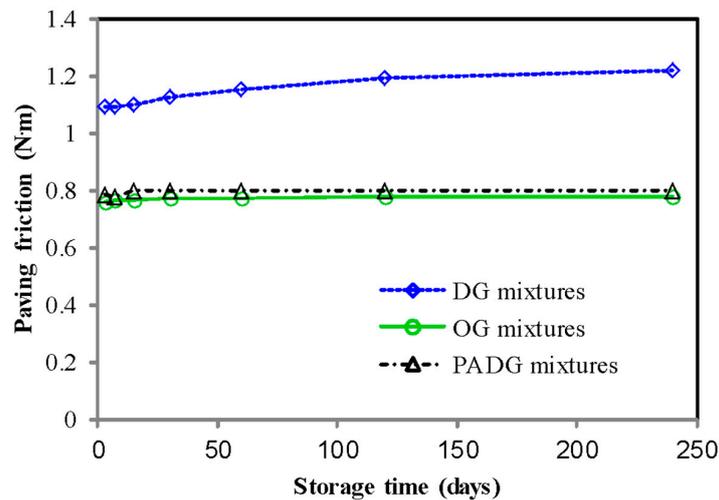


Figure 9. Paving frictions of cold mixtures at different storage times (at 5 °C).

3.3. Cohesion Analysis

As shown in Table 2, the percentages of retained materials were all larger than 60%, which is recommended from the Ontario Ministry of Transportation [1,8]. It indicates that three kinds of patching materials can meet the requirement of cohesion to alleviate the problems of raveling and stripping. Of them, the OG mixtures had the highest losing mass of 34.8 g, which increased by 1.83 and 0.37 times separately, when compared with the DG mixtures and PADG mixtures. This is because the continued gradation of the DG mixtures and the PADG mixtures is conducive to the denser compaction. The PADG mixtures have the highest residual ratio due to the cohesion enhancement of adhesive. The functional groups, with low surface energy in adhesive, can participate in the formation of the adhesive layer and reduce the surface energy between binder and aggregate, which will improve the infiltration ability of the binder [29].

Table 2. Results of cohesion tests.

Mixtures Type	Losing Mass (g)	Residual Ratio (%)
DG mixtures	12.3	98.46
OG mixtures	34.8	95.65
PADG mixtures	25.4	96.83

3.4. Stability Analysis

According to Mohr-Coulomb theory, the strength composition of the cold mixtures is essentially identical to that of hot asphalt mixtures. The strength composition depends on the cohesion from binder and the internal friction from the gradation and angularity of aggregates [30]. However, the strength formation of the cold mixtures has delay and hysteresis characteristics, as compared with the traditional hot asphalt mixtures. This is due to the existence of the dilute [3,31].

As seen in Table 3, it can be observed that the initial stability of the PADG mixtures was significantly greater than the DG mixtures and OG mixtures due to the reinforcement of the modifier and adhesive from the catalysis of the plasticizer from the break of the microcapsules during the compaction. However, DG mixtures and OG mixtures were added relatively more diluent to maintain an adequate workability. Moreover, there is not enough time for the diluent in the samples to volatilize fully at the low temperature of 5 °C, in the short fabrication process.

Table 3. Results of Marshall stability test in different conditions.

Mixtures Type	Stability in Different Conditions (kN)				
	Initial	Modeled	Low Temperature	Dry	Immersed
DG mixtures	0.30	5.54	4.40	5.32	3.80
OG mixtures	0.55	3.03	5.05	4.52	2.79
PADG mixtures	3.44	6.03	5.46	6.00	4.98

Molded stability was tested when most diluent was volatilized. Namely the binder cohesion was utilized to maximize the effect. As shown in Table 4, the PADG mixtures had the highest molded stability of 6.03 kN, which was slightly higher than that of the DG mixtures of 5.54 kN, but was 49.8% more than that of the OG mixtures. This problem could originate in the gap-graded gradation and bounding voids of the OG mixtures, which have a negative effect on the internal friction of the mixtures, creating poor interlocking structure.

Table 4. Results of freeze-thaw tensile strength tests.

Mixtures Type	Dry Tensile Strength (MPa)	Freezing-Thawing Tensile Strength (MPa)	Tensile Strength Ratio (TSR) (%)
DG mixtures	0.59	0.37	62.71
OG mixtures	0.43	0.25	58.14
PADG mixtures	0.62	0.50	80.65

In Table 3, it can be found that the stability of the PADG mixtures in low temperatures was larger than DG mixtures and OG mixtures by 19.4% and 7.5% in low temperature. This indicates that the PADG materials have a sufficient strength needed to withstand the failure or deformability during the winter. This attributed to the reinforcement of the soft segments from the modifier and continuous dense gradation of the PADG mixtures. Low-temperature deformation resistance and high temperature strength of mixtures can be effectively improved by the controlling ratio of the two segment contents.

Table 3 also presents two kinds of stability during dry and immersed conditions. It can be found that the PADG mixtures had the highest immersed residual stability ratio of 83.0%; however, the ratio of the DG mixtures and OG mixtures was only 71.4% and 61.7%. The modifier of the new polymer modified binder seems to improve the bonding of the PADG mixtures considerably even when in immersed conditions. However, the effects of the water on the other two mixtures are appreciable, due to the existence of more diluent in cold mixtures added to improve the workability. Furthermore, the gap-grade gradation of the OG mixtures contributes to numerous voids in the specimens, which exacerbates the seepage problems and is averse to the stripping resistance of the binder.

The time requirement for complete strength forming varies with the differences in grading and additives. Figure 10 describes the stability of three kinds of mixture samples, after fabrication and being stored in ventilated room condition at 5 °C to volatilize for 0.08, 3, 7, 15, 30, 45 and 90 days. It is clear that the stability of the PADG mixtures during the test process was always larger than the DG mixtures and OG mixtures, especially in 0.08 days. The increased speed of stability in the PADG mixtures was slower than the other two materials, but all three mixtures reached the steady state after 45 days. As discussed above, during the compaction process, the film capsules break, so the modifier and adhesive were cured rapidly in the early stages. However, the stability forming speed of the DG mixtures and OG mixtures is controlled by the evaporation rate of the diluent. It can also be found that the OG mixtures have the lowest stability in the three kinds of patching materials. This is because the PADG mixtures and the DG mixtures have continuous gradation, however, the gradation of the OG mixture is gap gradation. It causes a skeleton dense structure in PADG mixtures and DG mixtures to form, skeleton pore structure in OG mixtures to form.

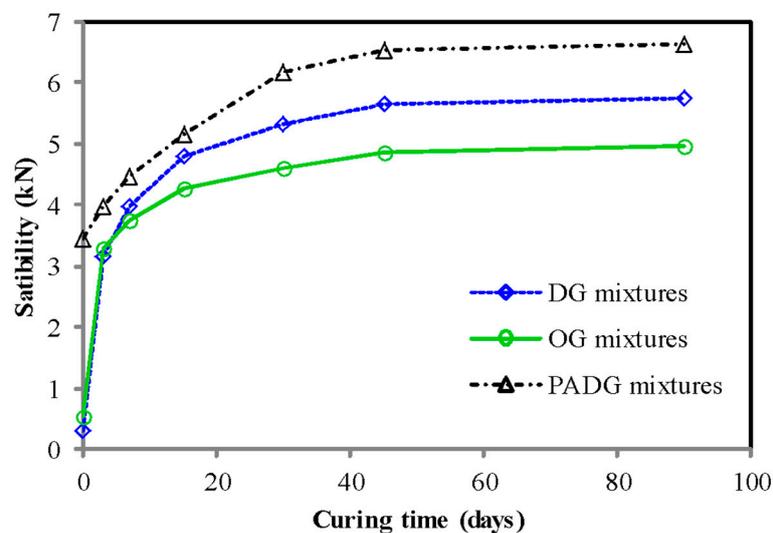


Figure 10. Stability of cold mixtures at different cured times at (25 °C).

3.5. Durability Analysis

3.5.1. Freeze-Thaw Tensile Strength Test Results Analysis

The ITS of the mixtures before and after the freeze-thaw and TSR of them is listed in Table 4. The PADG mixtures had the highest ITS of 0.5 MPa after freeze-thaw, followed by DG mixtures of 0.37 MPa and OG mixtures of 0.25 MPa. This indicates that the PADG mixtures still can approach the high level to bear traffic loads, even during adverse weather of low temperature and wet. The TSR of the PADG mixtures was larger than 80%, verifying that it provides satisfactory patching materials with insusceptibility to water and superior cohesion. However, the DG mixtures and the OG mixtures become fairly weak patching materials after a freeze-thaw, with a TSR of 62.7 and 58.1%, respectively.

3.5.2. Accelerated Loaded Test Results Analysis

It can be seen in Figure 11 that the dishing depths of the OG mixtures and PADG mixtures were smaller compared to the DG mixtures, when the test was performed in dry condition of 30 °C. The stronger bonding of the binder and the denser structure of the PADG mixtures could alleviate the deformation of the specimens under the loads. As for the OG mixtures, the high proportion of coarse aggregates can withstand the loads effectively, when in relation to the framework structure. It was also found that the dishing depth of all specimens increased sharply due to immersed treatment, especially the OG mixtures with a greater void content of 13.0%. In water immersion condition, a large number

of pores in the mixture will accelerate the water damage of the specimen. Among them, the PADG mixtures had the smallest dishing depth of 9.5mm, which had decreased by 84.2 and 142.1%, compared to the DG mixtures and OG mixtures, respectively, indicating that the modifier and adhesive can reinforce the strength of the binder in the PADG mixtures. Therefore, it can be concluded that the PADG mixtures have an excellent long-term performance even under traffic loading and adverse weather.

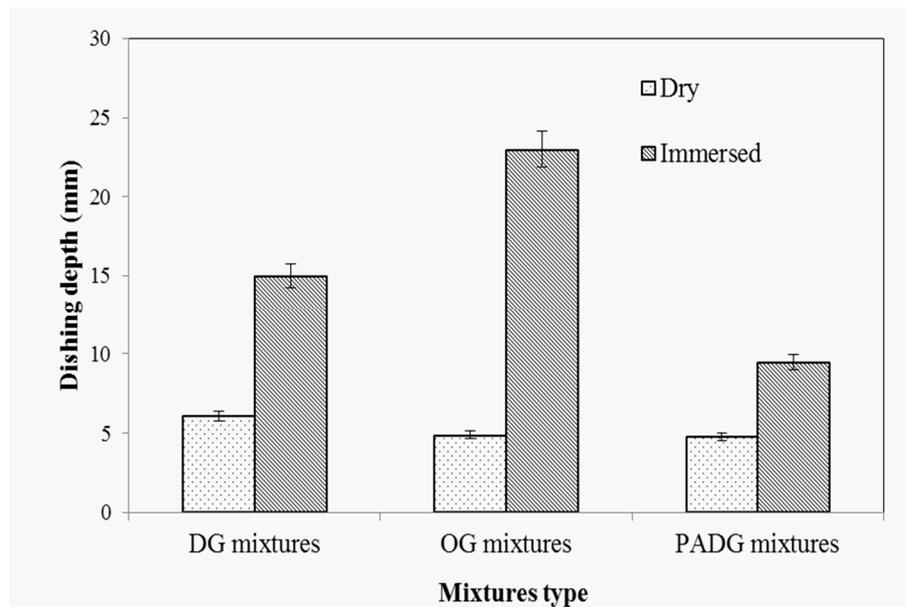


Figure 11. Accelerated loaded test results of DG mixtures, OG mixtures, and PADG mixtures in dry and immersed condition (at 30 °C).

4. Conclusions

This paper proposed new patching materials of polymer asphalt and dense-graded mixtures, which will ease the contradiction between the workability and the pavement performance in cold mixtures. It is based on the microcapsules' technique and polymer reinforcement of additive and dense gradation of aggregates. Laboratory tests and field surveys were performed to evaluate the performance when the cold mixtures were installed in low temperature and wet conditions. The main conclusions can be drawn as follows:

The performance of polymer asphalt and dense-graded mixtures was compared with the commonly used dense-graded mixtures and open-graded mixtures in the laboratory. These tests measured the workability, storability, cohesion, and stability durability of mixtures. The polymer asphalt and dense-graded mixtures had satisfactory workability and storability for the microcapsules alleviating the cured reaction, which were similar to the open-graded mixtures. All three mixtures had adequate cohesion to resistance releveling. Stability of the polymer asphalt and dense-graded mixtures had a significant advantage in the initial term, low temperature and immersion, as well as stability forming speed, which is meaningful for pothole patches to withstand the traffic load in adverse weather. The polymer asphalt and dense-graded mixtures had strong durability for the insusceptibility of water with a high TSR of 81% and low dishing depth when accelerated loaded in immersion.

Based on the above conclusions, it is believed that the polymer asphalt and dense-graded mixtures is practicable to apply for timely and durable pothole repairs in low temperature and wet conditions. Unfortunately, this paper only concentrated on the performance improvements of cold mixtures by microcapsules technique and polymer reinforcement in cold and wet conditions. Further studies involving properties of microcapsules in binder will be conducted at a later time.

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Conflicts of Interest: The authors declared that they have no conflicts of interest to this work. We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

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