Effect of Cationic Bitumen Emulsion Addition Time on Hydration of Cement

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Abstract—Bitumen emulsion in combination with Ordinary Portland cement is a commonly used additive in cold recycling applications. The current mix design approach for cold recycled mixes recommends the insertion of emulsion to a cement-reclaimed asphalt pavement material (RAPM)water mixture and suggests the mixing to be completed within one to two minutes of adding water to the cement. The current study investigates the impact of different emulsion insertion times on cement hydration and the engineering properties of the mixes. Samples used in the study were prepared using RAPM, cationic slow-setting bitumen emulsion (SS-2 grade), and Grade 43 ordinary Portland cement. Mixes were prepared using a constant Water-to-Cement (W/C) ratio of 1.5. Emulsion-to-Cement (E/C) ratio used for preparing the mixes was varied along with the time of emulsion insertion to evaluate changes in physicochemical properties of the mixes. Changes in hydration and engineering properties of mixes were evaluated using isothermal conduction calorimetry, X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, and Indirect Tensile Strength (ITS) tests, respectively. Observations from the study suggest that an increase in E/C ratio extends the breaking time of emulsion and delays the rate of strength gain in these mixes. Observations also suggest that the time window recommended in the current mix design approach may not be effective when E/C ratio is higher than 1.5. An insertion time greater than one minute, but less than the time where initial setting of cement starts may be effective in providing a higher strength (ITS) to cold recycled mixtures.

Keywords—bitumen cement interactions, delayed mixing of emulsion in cold recycled mixes, cement hydration, emulsion to cement ratio, bitumen emulsion, cement bitumen composite materials

I. INTRODUCTION

Bitumen emulsion in combination with Ordinary Portland cement is a commonly used additive in cold recycling applications like cold in-place recycling, cold inplant recycling, full-depth reclamation, etc. [1–4]. Cold recycling of bituminous pavements involves reusing RAPM to construct bituminous layers in pavement structures. In cold recycling, additives like bitumen emulsion and foamed bitumen are often used individually or in combination with cement to obtain the required structural properties in pavement layers [2]. The bitumen particles present in emulsion provide required flexibility to the mixture whereas the hydration of cement contributes to strength properties, facilitates reduction of early permanent deformation, and improves moisture resistance of emulsion-based mixtures [5, 6]. When compared to cement-based binders, strength gain in bitumen emulsion mixes is slower due to the longer time required for emulsion to break, cure, and bind the aggregate particles which in turn contributes to the strength properties of these mixtures. Hence, to accelerate the breaking process, cement is often recommended as an additive in emulsionbased mixes [7]. Cement hydration, pH increases in the mix, favorable surface charge of aggregates, etc. are some of the factors that contribute to early breaking of emulsion during curing period [8, 9]. However, the W/C ratio and bitumen-to-cement (B/C) ratio of RAPM mixes can vary depending on the type of application. The current study focuses on the impact of emulsion on hydration and strength gain processes of cement in the mixture.

Cement hydration is essentially an exothermic process that involves dissolution and precipitation of various ions (Ca²⁺, Na⁺, K⁺, H₂SiO₄²⁻, OH⁻, SO₄²⁻, Mg²⁺, etc.) to form hydrated cementing products like calcium-silicate-hydrate (C-S-H), calcium-aluminate-hydrate (C-A-H), etc. Reaction products like calcium hvdroxide (Portlandite/Ca(OH)₂), ettringite (AFt), monosulfate (AFm), etc. are also precipitated during the hydration process [10]. Researchers have used parameters like strength gain in mixes, heat of hydration, conductivity measurements, compositional changes of hydration products, etc. to evaluate the impact of bitumen emulsion on cement hydration. Studies have shown that cationic bitumen emulsion with a residual bitumen content of less than 10% by mass of cement has no impact on the kinetics of cement hydration [11]. However, an increase in residual bitumen content to nearly 20% was found to extend the dormancy period in cement hydration and slow down the rate of initial strength gain in cement-bitumen mixes [12-15]. Even though no appreciable differences in the heat of hydration were observed at the end of the 48 h, a higher B/C ratio was found to decrease the magnitude of cement hydration peak during early hydration periods and reduce the total heat of hydration in emulsion-based mixes [11, 12, 13, 15–20]. Bitumen in emulsion forms a thin film coating on the surface of cement particles which reduces the

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contact area between water and cement which in turn leads to a slower ion dissolution and a slower rate of cement hydration [18, 21]. Conductivity studies also support a slower ion dissolution in cement emulsion mixes during curing periods [12]. Electrostatic adsorption of positively charged bitumen droplets in cationic bitumen emulsion onto the negatively charged surface of tricalcium and dicalcium silicate phases (C3S and C2S respectively) in cement or adsorption of negatively charged droplets of anionic emulsion onto positively charged surfaces like tricalcium aluminate (C3A) in cement lead to the observed extension in dormancy period during cement hydration [15, 18]. The effect is more pronounced in the case of anionic emulsions when compared to cationic bitumen emulsions [18, 21, 22]. An increase in B/C ratio was also observed to decrease the workability of cement-bitumen emulsion mixes [17].

Studies in cement bitumen mortars have shown a higher concentration of cationic emulsion to reduce the Indirect Tensile Strength (ITS) in mixes during the initial curing period. However, with extended curing periods, the ITS values were found to be higher in these mixes when compared to mixes with a lower emulsion content [23]. Prior studies have shown that cement hydration products like Portlandite and ettringite from samples prepared using cationic emulsifier, water phase separated from cationic bitumen emulsion and Cement Bitumen Emulsion Pastes (CBEP) did not show any difference in diffraction peak locations (2θ values) when compared to hydration products precipitated in cement water slurries [24-26]. Even though the emulsion-bearing mixes did not show any new crystalline product formation during the hydration process, it was observed that the relative intensity of Portlandite diffraction peaks can decrease due to the presence of bitumen in these mixes [24, 26].

FTIR-based studies on Cement Bitumen Emulsion Paste (CBEP) samples have also shown that the precipitation of C-S-H phases can be inhibited in presence of cationic bitumen emulsion [12, 26]. Studies have shown that the FTIR spectrum of CBEP samples is a superposition of the spectra of cement powder, cured cement paste, and bitumen residue from bitumen emulsion and does not contribute to the formation of any new functional groups [26–28]. Moreover, an increase in B/C ratio was found to further reduce the precipitation of C-S-H phases during hydration process [12].

Even though existing studies have shown that emulsion can have a negative impact on cement hydration kinetics there are no studies available, to the best of our knowledge, that evaluate the influence of changes in emulsion insertion time (time when emulsion is added to cement-RAPM-water mixture) on hydration of cement. The present study looks into the effect of delays in bitumen emulsion insertion on hydration properties of cement present in the mix.

II. MATERIALS AND METHODOLOGY

A. Materials

100% RAPM was used in preparing Cold Recycled Mix (CRM) samples used in the study. RAPM for the study was

collected from a national highway section between Kanpur and Hamirpur, Uttar Pradesh, India. Parent material was found to have a bitumen content of 3.2%. The gradation of RAPM used in the study is given in Fig. 1.



Grade 43 Ordinary Portland Cement (OPC) was used as the additive to emulsion for preparing CRM samples used in the study. The oxide composition of cement estimated using X-ray fluorescence technique (WD-XRF, Rigaku make) and the physical properties of cement used in the study are given in Table I and II, respectively.

TABLE I. OXIDE COMPOSITION OF OPC

Composition	Percentage by mass of cement
Al ₂ O ₃	3.37
CaO	59.77
Fe_2O_3	3.89
K ₂ O	1.01
MgO	3.73
Na ₂ O	0.66
P_2O_5	0.09
SiO ₂	19.80
TiO ₂	0.77
NiO	0.00
SO ₃	2.39
MnO	0.06
L.O.I	4.4

TABLE II. PHYSICAL PROPERTIES OF OPC

Test parameter	Test result	Standard limits, IS 269- 2015 [29]
Specific gravity	3.12	-
Fineness, m ² /kg	301.7	225, minimum
Consistency, percent	29%	
Soundness, mm	2	10, maximum
Setting times		
Initial setting time, minutes	95	30, minimum
Final setting time, minutes	190	600, maximum
Compressive strength, MPa		
3 days	32.6	23, minimum
7 days	39.6	33, minimum
28 days	48.4	Between 43 and 58

Cationic slow-setting bitumen emulsion denoted as SS-2 in Indian standards, was used in the study. Details of the characterization of bitumen emulsion are given in Table III. Tap water was used in the preparation of the cement slurry

used in the study. The bitumen E/C ratio and W/C ratio used in sample preparation are detailed in Table IV.

Four different emulsion insertion times were evaluated in the study (a) no delay (cement, water, and emulsion added simultaneously and mixed immediately) (b) 1minute delay (cement and water mixed for 60 s followed by insertion of emulsion and the sample mixed for another 60 seconds), (c) emulsion added 60 minutes after mixing cement and water (d) emulsion added 120 min after mixing cement and water.

TABLE III. BITUMEN EMULSION CHARACTERIZATION TE	STS
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Test parameter	Test result	Standard limit, IS 8887-2018 [30]	
Particle charge	Positive	Positive	
Viscosity at 25°C by Saybolt Furol viscometer, s	35	30-150	
Residue on 600µm IS sieve, percent by mass	0.03	< 0.05	
Coagulation at low temperature	Nil	Nil	
Storage stability after 24h, percent	0.65	< 2	
Stability to mixing with cement, percentage	0.36	<2	
Miscibility with water	No coagulation	No coagulation	
Residue by evaporation, percent	64	> 60	
Penetration 25°C/100g/5 sec, dmm	84	60-120	
Ductility 27°C, cm	90	> 50	

For cases (c) and (d), slurry prepared using cement and water was kept inside a sealed container maintained at 30°C for respective time periods before emulsion was added.

 TABLE IV.
 Materials Proportions and Bitumen Emulsion Insertion Time

W/C	E/C	Delay in insertion of bitumen emulsion
1.5	1	
1.5	1.5	No delay, 1-min, 1-h, and 2-h
1.5	2	

A vortex mixer was used to mix the materials for calorimetry according to ASTM C1702-23e1 [31].

B. Methodology

1) Heat of hydration

TAM Air isothermal conduction calorimeter (TA instruments make) was used to measure the heat changes in samples during hydration process. Observations were made at a reference temperature of 30°C. Cement slurries needed for testing were first prepared in accordance with proportions specified in Table IV. Emulsion was then added to the slurry and mixed thoroughly before transferring the mixture to the calorimeter. Rate of heat flow was monitored for 72 h using the calorimeter and the total heat of hydration (cumulative heat released) was

estimated by integrating the heat flow rate curve over the designated time period.

2) Indirect tensile strength test

Indirect tensile strength test was performed to evaluate the changes in engineering properties of CRM prepared with different emulsion insertion times. Mixes meeting the required ITS criteria were first identified based on standard mix design process [32]. Mixes were prepared using 1% cement by weight of RAPM. Proportions of water and emulsion were then varied between 1% and 3% by weight of RAPM to identify the mix meeting the ITS criteria. Mix prepared using 1% cement, 1.5% water, and 1.5% emulsion was selected based on the mix design and used for evaluating the impact of changes in emulsion addition time on RAPM mixtures. To evaluate the impact of delay in emulsion addition, RAPM and cement were dry mixed (identified as a dry mix) before adding water and emulsion. Mixing conditions were kept identical to those used in preparing cement-water-emulsion slurries for estimating the heat of hydration. (a) For the mix with no delay, water, and emulsion were simultaneously added and mixed with the dry mix (b) For the scenario involving 1-minute delay, water was mixed with the dry mix for one minute before adding emulsion (c) For samples with delays of 1 and 2 h, water was mixed with the dry mix and the samples were stored in an airtight container at ambient temperature for the respective durations before mixing with emulsion.

Six cylindrical samples of 101.6 mm diameter and 63.5 mm height were prepared for each CRM in accordance with the Marshal compaction technique [6]. Specimens were extracted from the mold 24 h after compaction, cured in an oven at 40°C for 72 h, and subsequently cooled to room temperature. Samples for each mix were divided into two groups (3 nos. each) for dry and moisture conditioning, and ITS was conducted using Marshall apparatus at a loading rate of 50 mm/min following South African guidelines [32].

3) XRD and FTIR analysis

XRD and FTIR Spectroscopy techniques were used to identify new compounds or changes in mineralogical characteristics of cement hydration products in presence of bitumen emulsion.

CBEP samples for XRD and FTIR testing were cured for one day at ambient temperature and treated with ethanol to arrest further hydration. Samples were then treated with trichloroethylene (TCE) to remove bitumen and crushed gently using a mortar and pestle [20]. Crushed CBPE samples were then dried at 40°C for 24 h (until no weight change was observed in samples) and stored in a desiccator for further testing. Sub-samples for XRD and FTIR analysis were randomly selected from the stored samples.

An X-ray Diffractometer (MiniFlex-600, Rigaku make) was used to perform XRD measurements. Samples were tested at 2θ range of 5° to 70°, 0.02° step size, at a scan speed of 3° per minute. American Mineralogist Crystal Structure Database (AMCSD-RRuff) and Crystallography Open Database (COD) were used as references in identifying minerals in cement powder and hydrated cement paste. Fourier Transform Infrared Spectrometer (FTIR, Perkin Elmer make) with attenuated total reflection

(ATR) mode and scan range of 400 to 4000 cm⁻¹ was used for FTIR measurements.

III. RESULTS AND DISCUSSIONS

A. Heat of Hydration

Heat flow rate and cumulative heat released in samples after mixing were monitored for 72 h and given in Figs. 2 to 4 and Fig. 5 to 7.



Fig. 2. Heat flow rate curves of cement paste and CBEPs with E/C=1.

Observations in Figs. 2 to 4 clearly show the addition of emulsion to delay cement hydration. Observed peak shift and reduction in intensity of main hydration peak, and extension of dormancy period can be attributed to delayed cement hydration in the matrix possibly due to formation of thin bitumen film over cement particles that inhibit accessibility of water to cement particles [11, 18, 20, 21, 24].



Fig. 3. Heat flow rate curves of cement paste and CBEPs with E/C=1.5.

When compared to samples with no delay in mixing emulsion and cement paste, increasing mixing time by up to one hour was observed to speed up the rate of cement hydration in mixes (Figs. 2, 3, and 4). Initial setting and hardening of cement paste may be influencing the mixing of paste and emulsion in the case of samples where emulsion addition was delayed by two h as reflected in Fig. 2 and 3.

However, an increase in bitumen concentration with respect to cement in the mixture (Fig. 4) was found to increase the delay in cement hydration [17]. However,

when emulsion concentrations are higher (Fig. 4), delaying the inclusion of emulsion was found to enhance the rate of heat flow in these mixes. Cumulative heat of hydration in samples was estimated and given in Figs. 5 to 7. Observations show that the total heat release from CBEP samples to be lower than that of cement paste during the initial curing periods (24 to 32 h).



Fig. 4. Heat flow rate curves of cement paste and CBEPs with E/C=2.

However, the cumulative heat release in CBEP mixes was found to be higher than that of the cement paste after 72 h of hydration [33]. Heat energy released during breaking of emulsion is possibly contributing to the observed behavior in these samples.



Fig. 5. Total heat of hydration curves of cement paste and CBEPs with E/C=1.



Fig. 6. Total heat of hydration curves of cement paste and CBEPs with E/C=1.5.

Similar to observations in Figs. 2 to 4, delaying the insertion of emulsion in CBEP mixes was found to have a

higher impact when bitumen concentration in mixes increases (increase in E/C ratio).

Considering the changes in slope of cumulative heat release in CBEP mixes, it is reasonable to assume that the breaking of emulsion starts at around 36 to 40 h after bitumen insertion into the matrix.



Fig. 7. Total heat of hydration curves of cement paste and CBEPs with E/C=2.

Furthermore, for mixes prepared using the current mixing approach (up to one minute of mixing time) breaking of emulsion was found to get delayed with an increase in emulsion concentration (increase in E/C ratio). Hence, it may be inferred that a higher heat release in mixes with delayed emulsion insertion expedites the breakage of bitumen present in the emulsion (Figs. 5 to 7).

B. Indirect Tensile Strength Test (ITS)

Impact of different emulsion insertion times on engineering properties of mixes was evaluated based on ITS test and given in Table V. Specimens for ITS testing were prepared at an E/C ratio of 1.5 where emulsion insertion times are varied in accordance with details discussed in section (II-B-2). Observations in Table V suggest that delaying emulsion insertion by 1 min increases the dry ITS value by nearly 35%. Uniform coating of cement pastes on RAPM aggregates achieved during one minute of mixing may be causing the higher strength observed in these mixes.

TABLE V. ITS RESULTS OF DRY AND MOISTURE CONDITIONED SPECIMENS WITH E/C OF 1.5

Delay in bitumen emulsion addition time	ITS-dry, kPa	ITS-wet, kPa
No delay	167±04	137±14
1 min	225±09	192±15
1 h	164±10	115±10
2 h	172±05	119±15

Delaying emulsion insertion by 1 h or above was found to have a minimal benefit on ITS values when compared to the case where water, cement, and emulsion were mixed simultaneously. Considering the strength increase with one-minute insertion delay and assuming that initial setting of cement can occur within the extended time (one to two h) provided in other cases, it is reasonable to assume that providing the cement paste hydration time of 10 to 15 min may generate additional heat required for early breaking of emulsion and also provide higher ITS values for the mixtures. However, this needs to be further verified through additional testing of these mixes. ITS estimation of these cases in mixes with E/C ratios of 1 and 2 may also be required to confirm the above hypothesis.

C. XRD Analysis

Presence of cement hydration products like Portlandite and ettringite were analyzed using XRD to verify the impact of emulsion insertion time on cement hydration and given in Fig. 8 to 10. XRD data of pure and hydrated cement paste after one and three days of curing are given in Fig. 8. Results in Fig. 8 show the intensities of alite (C₃S) and belite (C₂S) peaks to decrease and the peaks of tricalcium aluminate (C₃A), gypsum (CaSO₄·2H₂O), and bassanite (CaSO₄·0.5H₂O) to disappear with three days of curing [34, 35].



Fig. 8. Comparison of XRD pattern of cement powder with 1-day and 3-day cured cement paste.



Fig. 9. Comparison of XRD pattern of 1-day cured cement paste with 1-day cured CBEPs with E/C=1.

Results in Fig. 8 also show a steady increase in intensity of Portlandite (Ca(OH)₂) peaks and emergence of ettringite peak during hydration period. Observations suggest that the pozzolanic reactions are continuing in the material resulting in increased concentrations of Portlandite in the matrix. Depletion of tricalcium aluminate and gypsum also suggests that the allied reactions during cement hydration are also progressing unhindered in the matrix [34, 35]. Observations on one day cured CBEP samples with an E/C ratio of 1.0 and subjected to various bitumen insertion times are given in Fig. 9.



Fig. 10. Comparison of XRD pattern of 1-day cured cement paste with 1-day cured CBEPs with E/C=2.

observed by earlier researchers, minerals As precipitated during cement hydration did not show any peak shifting behavior due to insertion of emulsion suggesting the absence of crystallographic changes in the reaction products [26]. However, differences in peak intensities with varying test conditions suggest changes in material concentrations across these samples. Observations in Fig. 9 clearly show that the intensity of Portlandite peak in CBEP mixes with no delay in emulsion insertion to be less than those observed in pure cement pastes. Hence it is reasonable to infer that the hydration of cement paste is hindered when emulsion is added prior to the start of cement hydration. However, in mixes where there is a delay of one minute in adding emulsion, Portlandite concentrations are seen to be higher than samples with zero delay, and lesser than quantities observed in pure cement paste. Furthermore, the Portlandite peak intensities are found to decrease once emulsion insertion is delayed for one and two h suggesting the precipitated Portlandite to be used up in pozzolanic activities. A comparison of XRD peaks in Fig. 9 also suggests that the progress of cementitious reactions is slower in case of CBEP samples where emulsion insertion is delayed by one minute. Observations on ettringite precipitation peaks and the cumulative heat generated in samples (Fig. 5) also support the above observations. XRD results of one day cured CBEP samples with an E/C ratio of 2.0 when subjected to different bitumen insertion times are given in Fig. 10.

Comparison of XRD peaks of cement paste, CBEP with no delay, and CBEP with one minute delay suggest that an increase in emulsion content can hinder hydration of cement in these mixes (Fig. 9 and 10). The above observations align well with the cumulative heat release data given in Fig. 7. When compared to samples with an E/C ratio of 1.0, Portlandite precipitation was found to be significantly lower in samples with a higher amount of emulsion when emulsion was added along with water and cement (no delay case). A higher concentration of Portlandite observed in CBEP 1-hour and 2-hour delay samples suggests a delayed emulsion to be beneficial in mixes with higher E/C ratios (Fig. 10). Comparison of ettringite peak intensity among samples with E/C ratios 1.0 and 2.0 also supports the role of bitumen in retarding cement hydration in CBEP mixes (Fig. 9 and 10).

D. FTIR Spectroscopy Analysis

Presence of cement hydration products like C-S-H, Portlandite, ettringite, and monosulfate was further verified using FTIR technique and given in Fig. 11 and 12. FTIR results of cement powder and hydrated cement pastes after one and three days of curing are given in Fig. 11. Observations suggest that the sulfate available in cement (wave number 1161) transforms into monosulfate (AFm) phase (wave number 1112) after 3 days of curing. Si-O asymmetric stretching peaks (wave number 919) from alite (C₃S) and belite (C₂S) phases were observed to be replaced with calcium silicate hydrate (C-S-H) peak (wave number 962) suggesting progressive cementitious reactions in the matrix [33–36].



Fig. 11. Comparison of FTIR spectrum cement powder with 1-day and 3-day cured cement pastes.

FTIR data of CBEP samples with varying E/C ratio and cured for one day are given in Fig. 12a and 12b. Spectral comparison of cement paste and CBEP samples shows no shift in peak positions due to the insertion of emulsion. Observation is indicative of the absence of any chemical changes in cement hydration products due to bitumen insertion in the matrix [12, 26].

Peak signals in Fig. 12a and 12b were normalized against O-H-O bending peak observed at wave number 1644 which did not show any change in intensity during emulsion addition and curing. Observations in Fig. 12a and 12b clearly suggest that the intensity of monosulfate (AFm) phases (wave number 1112) to increase with delay in bitumen insertion. Observations suggest a higher sulfate utilization in the mixes toward formation of ettringite which transforms into a monosulfate phase as hydration period progresses. Lesser quantity of monosulfate formed in mixes with zero or 1-minute delay in emulsion addition also supports the above hypothesis and validates the role

of bitumen in retarding cement hydration process. Silica tetrahedron peaks corresponding to formation of C-S-H phases (wave number 962) also show a similar trend when emulsion insertion time varies in CBEP mixes. Comparison of data in Fig. 12a and 12b (E/C ratio=1.0 and E/C ratio=2.0 respectively) also shows the intensities of respective peaks to be lower in mixes with an E/C ratio of 2.0 when emulsion was added after 1-min delay. However, the difference in peak intensities was minimal once the delay in bitumen insertion was one hour or more. Comparison of data given in Fig. 12a and 12b agrees well with observations of previous researchers where the presence of bitumen was found to retard cement hydration [12].



Fig. 12. Comparison of FTIR spectrum of 1-day cured cement paste with 1-day cured CBEPs with (a) E/C=1.0 and (b) E/C=2.0.

IV. CONCLUSIONS

Based on the analysis of the heat of hydration, ITS results, XRD patterns, and FTIR spectra, the following inferences were drawn regarding the effect of bitumen emulsion addition time on cement hydration:

Bitumen present in emulsion can form a thin layer over cement particles present in CBEP which negatively influences the hydration of cement in these mixes. An increase in emulsion/bitumen content with respect to concentration of cement (E/C ratio) can extend the delay in breaking of emulsion which is critical in the mix attaining the required strength properties.

The current approach followed in preparation of CRM where mixing of emulsion with cement-RAPM-water mix is recommended to be completed within a minute may not be effective in case where the E/C ratio is higher than 1.5.

Observations suggest that a mixing time longer than one minute and less than the time when the initial setting of cement starts (possibly 10 to 15 min) may be effective in providing a higher strength (ITS) to CRM mixes. Additional testing on samples prepared using the suggested mixing times is required to validate the above observation.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Suryakapa Jaipal Reddy worked on methodology, testing, data collection and analysis, writing-original draft preparation; Syam Nair worked on conceptualization, supervision, writing-reviewing and editing; all authors had approved the final version.

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