Enhanced near-infrared reflectance and functional characteristics of nano metal oxide embedded alkyd coatings

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Abstract

The main idea of the research is to formulate alkyd based smart coatings with high NIR-reflectance to decrease the cooling load in order to maintain cool comfort in buildings. In addition to the NIR reflectance studies, the physical characteristics of the coating such as glossiness, hiding power, spreading area, volatile organic contents are evaluated. Heat reflective coatings are prepared by ball milling technique utilizing white metal oxide nanoparticles (NPs) titanium oxide (TiO₂), zinc oxide (ZnO) and zirconium oxide (ZrO₂) as pigments. The x-ray Diffraction (XRD) and Field Emission Scanning Electron Microscopy (FESEM) results reveal the prepared metal oxide pigments are of high purity. Bonding interaction between the alkyd resin and the NPs are studied using Fourier transform infrared spectroscopy (FTIR). The spectral reflectance of the prepared coatings are measured using spectrophotometer in the solar spectrum range, wherein ZnO based nanocoatings shows better results. The cool comfort is measured for ZnO nanocoatings in an enclosed glass cabinet and the differences in temperature with respect to exposure to sunlight are highlighted. Finally, a prototype coated with ZnO NPs maintains lower indoor temperature than the commercially available white paint coated model in the order of 2.7 °C.

1. Introduction

The world is facing acute global climate change because of release of large quantities of greenhouse gas emissions. Energy saving process is one of the important research topics now-a-days which may reduce those emissions. The sun’s energy that reaches the earth’s surface are classified as Ultraviolet Region (300–400 nm) about 5%, Visible Region (400–700 nm) around 43% and Infrared region (700–2500 nm) 52% [1, 2]. The heat producing region of the infrared radiation ranging from 700 nm to 1100 nm [3] is responsible for the warm nature of open air. Coatings colored with conventional inorganic pigments absorb these invisible near-infrared radiation (NIR) and sustains warm condition inside the building too. In this aspect, the technology called cool-color roofing utilizes solar reflective pigments in the form of coatings on to the windows, house roofing, walls of buildings, etc. These pigments reflects the Near-infrared (NIR) radiation and maintain cool comfort in the interior thus minimizes consumption of energy for cooling interiors [4].

The NIR reflective pigments are broadly classified as inorganic and organic pigments. They reflect wavelengths in the infrared region and also, reflect some visible light selectively. The inorganic classes of NIR reflective pigments are mainly metal oxides and they are increasingly used for roof and building coatings because of their excellent weather abilities and high heat stabilities [5–13]. Their applications are not limited to architectural, construction coatings, tiles, plaster etc, but also used in automotive exterior and interior application like dash-boards, fuel tanks etc. In addition to the studies relating to the metal oxide based NIR reflective pigments there are numerous research reports dealing with the use of metal ion doped metal oxide NIR reflective pigments in coating industry to enhance the reflective properties for cool comfort [14–16]. In this context, various research activities have been carried out in recent decade but many of the coating are not
formulated in the alkyd resin. Meenakshi et al (2018) [17] prepared bismuth titanate (BTO) cool pigment and studied the IR reflectance property. Thermal studies of BTO dispersed in acrylic coated steel substrate reduce the interior temperature by almost 10 °C. Chalakorn Chaowanapanit et al (2020) [18], investigated the energy saving nature of a paint compared with conventional paint. They found that high solar reflective paint could reduce exterior surface temperature by as high as 8.1 °C and save energy by 31.24% from decreasing cooling load. B Xiang et al (2017) [19] evaluated the cooling properties by incorporating barium titanate in acrylonitrile-styrene-acrylate terpolymer which shows a highest reflectance value of 67.66%. Also the studies proved that the improvement in the cooling properties does not affect the mechanical properties of the coating. Zhilang You et al (2021) [20] prepared CuO microparticles (MPs), CuO NPs & TiO2 NPs and formulated epoxy based coatings and studied their spectral reflectances. They found that the spectral reflectance of the CuO NPs and TiO2 NPs blended coating is higher than that of the CuO MPs. Green coloured Y2BaCuO5 nano-pigment was used as cool pigments by coating over building roofs [21]. The NIR reflectance efficiency of (polymethyl methacrylate) PMMA polymer matrix composites reinforced with different size ZnO particles were studied and reported that the morphology of pigment also plays a significant role to obtain high NIR reflecting property [22]. Song et al (2013) [23] developed cool white coatings for building envelopes for improving building energy efficiency. They found that the usage of cool white coating significantly reduced the radiant heat flux inside the building.

In this context, it comes out that adequate passive building technologies contribute to mitigate the cooling demand increase, and improving the thermal comfort in non-cooled buildings without compromising the physical aesthetic appearance of the coating. To address this, the physical characteristics such as gloss level, spreading area, hiding power, and solid content of the prepared coatings were evaluated. Though many studies are with the metal oxide NPs, utilization of nano sized white pigments in the formulation of alkyd coatings is unexplored. It is well known that white pigments present high near-infrared (NIR) reflectance [24, 25]. Alkyd resins are extensively used in coating industry as one of the major ingredients in many synthetic paints, enamels, varnishes, primers, etc, which are used for coating range of materials like wood, metal, plastic, composite, etc with a good gloss retention. The objective of the pursued work is to formulate a NIR reflective white alkyd coating utilizing TiO2, ZnO, and ZrO2 white pigments prepared by chemical route. The structural and morphological properties of the pigment were discussed using XRD and FESEM respectively. The NIR reflecting...
properties of the nano-formulated coatings (NFCs) were estimated from NIR reflectance spectroscopy. A glass model was fabricated and coated with the optimally best NIR reflecting coating to study the heat reflecting effect. In addition to this the wetting ability and corrosion studies were also done.

2. Materials and methods

All the precursors used in the preparation of metal oxide nanoparticles were purchased from Merck Chemicals Ltd, India. The additives used in the preparation of metal oxide coatings were used as such received from the suppliers: Linseed alkyd resin (Sunny Paints and Tar Products, India), Thickener A (Vigneshwara Paints Ltd, India), Soya lecithin (Shreenidhi Oils and Foods Ingredients Pvt. Ltd, India), toluene, aluminium stearate and cobalt naphthenate (Sigma Aldrich, India). TiO₂, ZnO, and ZrO₂ NPs used in this study were synthesized by sol-gel, hydrothermal and gel combustion method respectively by following the procedure reported elsewhere [26–28]. Briefly, the preparation of nanomaterials is discussed below.

2.1. Preparation of nanomaterials

Titanium oxide nanoparticles are synthesized by Sol-Gel method and the reaction process is schematically shown in figure 1. 100 ml of isopropyl alcohol is added to 15 ml of titanium tetraisopropoxide. It is stirred in a magnetic stirrer for about 10 min 10 ml of deionized water is added drop wise and stirred for about 24 h. After that a gel formation takes place, the gel is collected and dried at 100 °C in a hot air oven for 3–4 h. The dried gel powder is then calcinated at 500 °C for a period of 1 h produces TiO₂ nano particle.

Zinc oxide nanoparticles are synthesized by hydrothermal method as depicted in figure 2. Zinc acetate (2.195 g) dissolved in 50 ml of distilled water is mixed into the solution containing 4 g of ammonium oxalate with stirring. The pH of the solution is raised to 10 by adding ammonia solution. Within a short span of time, white precipitate of zinc oxalate is formed. The formed precipitate is then filtered and washed. The filtered precipitate is placed in a hot air oven at 105 °C for a period of 1 h. It is then collected in a clean silica crucible and then heated at 700 °C for 1 h in the muffle furnace, leaving white colour zinc oxide nanoparticles.

Zirconia nanoparticles are synthesized by gel combustion method and the process flow diagram is shown in figure 3. 4 g of Zirconium nitrate and 4.2 g of citric acid are dissolved in 100 ml of distilled water and stirred continuously. The pH of the solution is slowly raised to 10 by adding ammonia. The obtained sol is heated at 80 °C in a hot plate with continuous stirring. After certain period of time, self-combustion takes place. The formed ash is collected and calcinated in a furnace at 700 °C–800 °C for a period of 2 h, which produces the ZrO₂ nanomaterials.
2.2. Formulation of alkyd based nanocoating

The nano-formulated coating was prepared by mixing the prepared metal oxide NPs as pigment and the alkyd resin as binder along with other additives as per our earlier report [29]. The mixing of the various components was done by ball milling technique where the materials in various wt% were milled for a period of 6 h in a tungsten carbide bowl by keeping the ball to powder ratio as 10:1, at a speed of 300 rpm. After milling, the nano-formulated paint was transferred into an airtight container and used for further characterization. The coatings are shortly designated using abbreviations as TOC (titanium oxide based coating), ZOC (zinc oxide based coating), and ZrOC (zirconium oxide based coating). Quality of NFCs were evaluated by obtaining a dry pigmented film developed over a glass slide using brush coating method followed by air drying.

3. Results and discussion

3.1. Characterization of NPs

3.1.1. XRD analysis

The NIR reflectivity depends on the structural parameters of the pigments, it is essential to evaluate the crystallite size of as obtained NPs. XRD is used for the assessment of phase purity and crystallinity of materials. XRD patterns were acquired from a Rigaku Ultima IV automated multipurpose x-ray Diffractometer (USA) with Cu Kα radiation (λ = 1.5418 Å) operating at 40 kV having 30 mA current. Data were acquired in the range of 2θ = 20°– 80° and the XRD peaks obtained for prepared NPs are shown in figures 4(a)–(c). The XRD of TiO2 NPs (figure 4(a)) display their characteristics peaks at 2θ = 24.83°, 37.54°, 47.78°, 53.98°, 54.61°, 62.89°, 68.25°, 74.84° are assigned to the miller indices of (101), (004), (200), (105), (211), (204), (116) and (107) respectively, well resembled with the JCPDS.No.89-4921. The structure is found as tetragonal with body-centered cubic lattice [30]. The XRD pattern of ZnO NPs shows the characteristic diffraction peaks at 2θ = 31.35°, 33.82°, 35.83°, 37.60°, 43.75°, 47.66°, 76.55° with the miller indices of (100), (002), (101), (102), (110), (103), (200), (112), (201), (202) lattice planes respectively as per the JCPDS.No.89-1397 data, exhibiting a hexagonal structure (figure 4(b)) [27]. The diffraction peaks of ZrO2 NPs shows prominent peaks at 2θ = 30.33°, 34.77°, 45.51°, 50.53°, 56.52°, 59.96°, 66.26° and 75.37° (figure 4(c)), all these peaks are indexed to the lattice planes of (111), (200), (220), (311), (222), (400) and (420) respectively [JCPDS No. 49-1642] [31] having cubic system with face centred lattice. The crystallite sizes of the prepared NPs are evaluated using the Debye-Scherer formula and found to be 35 nm, 32 nm and 22 nm for TiO2, ZnO and ZrO2 NPs respectively.
3.1.2. FESEM analysis
FESEM helps to examine the surface morphology and microstructure of the obtained NPs. FESEM images are recorded through FESEM-Carl Zeiss MA15/EVO 18 electron microscope (Germany). Figure 5(a) represents the FESEM image of TiO$_2$ NPs which are agglomerated type with ranges below 100 nm. The ZnO NPs prepared by precipitation technique shows well distinguished and defined structure owing to the high degree of crystallinity [figure 5(b)]. The sizes of the particles are in the range between 100–150 nm. Figure 5(c) shows FESEM image of ZrO$_2$ NPs. It is also like an agglomerated one, owing to the use of pectin for the preparation. Also, the size of the NPs ranges in between 100–200 nm.

3.2. Characterization of NFCs
The coatings formulated using NPs as pigment and alkyd resin as binder is subjected to various test such as gloss measurements, spreading area, hiding power, and solid content to access the quality of the prepared NFCs. Also, the molecular vibrations present in the coating were evaluated using FTIR spectra. Further, the anticorrosion ability, wetting ability and NIR reflectance of the NFCs were evaluated.

3.2.1. Physical characteristics
The shining appearances of the NFCs were evaluated from the gloss measurements. The glossiness is calculated by directing a visible light towards the coated surface and the reflected light was captured at 60° angle. The intensity of the reflected light was correlated to the gloss level by an integer. The reflected light intensities of the various coatings are summarized in table 1 and the gloss levels were correlated for the coating according to the ASTM standards (D523-14). The NFCs show high degree of glossiness with gloss level higher than 6 indicating its shiny nature. Among the NFCs, the TOC was found to have gloss unit of 89 indicating maximum gloss finish.

Spreading ability of NFCs were tested by coating 1 g of paint over a draw down card by slowly pointing from top to down at a slow rate [ASTM D 344-11]. After the application, the coated surface was exposed to air for about 24 h at 300 K for drying. The area covered by the different films were evaluated and given in table 1. It is found that ZOC has a spreading area of 72.74 cm$^2$ which is maximum among the NFCs. Such a high spreading area obviously minimizes the consumption of paint for the application purpose.
The drawdown card possesses black and white background, from which the hiding power can be calculated. The coating possessing good hiding power masks the entire background on which the NFCs are applied. The hiding power of the NFCs were calculated as per the ASTM standard using the formula

\[ H = \frac{A \times N \times D}{10M} \quad (1) \]

where, \( A \) is the film area coated by the paint, \( N \) is the fractional Non-Volatile or the solid content of the formulated coating, \( D \) is the density of the paint and \( M \) is the dry weight of the draw down card. Figure 6 displays the optical images of NFCs after applying over the draw down card. From the image, it is clear that the prepared paints were able to protect both black and white background of the drawdown card. From the tabulated values [table 1], ZOC possesses high hiding power compared to other NFCs.

The volatile organic content (VOC) present in the coating was determined by adopting the standard ASTM D 5201–05a. 1 g of paint was dried in hot air oven at 110 °C for 1 h taken in a clean watch glass. The percentage of non-volatile content was calculated using the following equation (2),

\[ \% \text{of non-volatile content} = \frac{\text{Mass of dried paint residue}}{\text{Mass of Wet paint sample}} \times 100 \quad (2) \]

Normally, solvent based paints complying with the regulatory limits of 52 % of solid content are considered as high solid content paints. As per the experiment conducted, the non-volatile content available in TOC, ZOC and ZrOC was found to be 62%, 62% and 52% respectively. The high solid content facilitates high coating thickness with the reduction in emission of VOC, reduction in the application cost, consumption of paint, etc.

| Sample | Color of the coating | Glossiness units | Gloss level | Spreading area A (cm²) | Hiding power (m² L⁻¹) | Percentage of non-volatile content (%) |
|--------|----------------------|------------------|-------------|------------------------|-----------------------|--------------------------------------|
| TOC    | White                | 89               | 7           | 64.815                 | 1.855                 | 62                                   |
| ZOC    | White                | 86               | 7           | 72.74                  | 2.07                  | 62                                   |
| ZrOC   | White                | 78               | 6           | 71.05                  | 1.66                  | 52                                   |

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3.2.2. FTIR analysis of NFCs

FTIR studies were done using Bruker Optics, Germany to ensure the chemical identity of NPs and the NFC, with an average of 24 scans. Figure 7(a) represents the FTIR spectrum of alkyd resin. It exhibits characteristic vibration for C–O bonds (1070 cm\(^{-1}\)), C–H in-plane bending vibrations (1121 cm\(^{-1}\)), C–O–C group vibrations (1261 cm\(^{-1}\)), a pyrrole ring (1459 cm\(^{-1}\)), C–C Stretching vibration (1600 cm\(^{-1}\)), a carboxylic group vibration (1730 cm\(^{-1}\)) and C–H vibrations of a methyl and methylene groups (2800–3000 cm\(^{-1}\)). Figures 7(b)–(d) represents the FTIR spectrum of the TOC, ZOC, and ZrOC respectively. In addition to the mentioned peaks for alkyd resin, some additional peaks are observed in 550–700 cm\(^{-1}\) region corresponds to the presence of metal oxide. In TiO\(_2\) dispersed alkyd coating 517 cm\(^{-1}\) is attributed to the Ti–O stretching vibration. Similarly, ZnO and ZrO\(_2\) dispersed coating exhibit their characteristic vibration at 568 cm\(^{-1}\) and 552 cm\(^{-1}\) confirm their existence metal oxide in the NFCs.

3.2.3. Film thickness by AFM

The paint film obtained by coating the NPs dispersed alkyd resin over glass substrate normally forms a very thin film like coating of 3–5 \(\mu\)m thickness. In this work, AFM technique is used to elucidate the thickness of the dry pigmented film obtained after applying over a glass substrate of 7.5 cm \(\times\) 2.5 cm specimen. The cantilever of the
AFM is adjusted to scan the interface of the film where painted and uncoated region found on the glass substrate. About $2.5 \, \mu m \times 2.5 \, \mu m$ of the area covered the coated and uncoated position is scanned during imaging. Figures 8(a)–(c) represents the 2D-AFM image of the coated (light brown colour)/non coated segment (dark brown colour) scanned by the cantilever. From the line profile analysis [figures 8(d)–(f)], the thickness of the TOC, ZOC, ZrOC are found to be 4 $\mu m$, 4 $\mu m$ and 3.5 $\mu m$ respectively. Owing to the dispersion of NPs, the obtained film coating is also in lesser thickness ($3–4 \, \mu m$).

3.2.4. Water contact angle measurements

Water Contact Angle (WCA) measurement was performed using Data physics Instruments, Germany with a dosing volume of 5 $\mu l$ at a dosing rate of 1 $\mu l \, s^{-1}$. Surface tension is an influential parameter in characterizing the wetting and adhesion phenomena of the coated surfaces. In order to evaluate the adhesion of coated film, it is necessary to evaluate its surface tension values. To form a strong adherent coating on a chosen substrate, the paint suspension obtained should possess least critical surface tension and then only it can spread and adhere well on the substrate easily. The coating with least surface tension after drying process produces the pigmented film and also exhibits the critical surface tension in lower level. Based on the water contact angle values measured using goniometer as per the standard ASTM D7734 using three base fluids namely water, isopropanol and ethylene glycol, the critical surface tension of the NFCs are computed using William-Zisman plot shown in the figure 10.

The obtained $\gamma_{c}$-critical surface tension of the film is in least range of 15.72 mN m$^{-1}$, 15.05 mN m$^{-1}$, and 15.87 mN m$^{-1}$ for the TOC, ZOC and ZrOC respectively. A lower WCA reveals a strong wetting ability and helps in improving the spreading rate, thereby increasing the surface contact, adhesion and coverage area of the paint.

3.2.5. Corrosion protection ability of NFCs

3.2.5.1. Wet corrosion studies

The stability of NFCs under acidic conditions is examined using the acid immersion tests. The corrosion inhibition efficiency of the coating is determined from weight loss method and is depicted in figure 11. The percentage of weight loss of the coated steel displays remarkably reduced values in 0.1 N HCl, 0.1 N H$_2$SO$_4$ and 0.1 N HNO$_3$ environments on comparing with bare steel (figure 11). From the weight loss values, the corrosion inhibition efficiency (%) of TOC in 0.1 N HCl, 0.1 N H$_2$SO$_4$ and 0.1 N HNO$_3$ is calculated as 96.14%, 87.88% and 97.46% respectively. The inhibition efficiency of ZOC is found as 96.31%, 86.89% and 97.65%. Similarly, the inhibition efficiency of ZrOC is 95.91%, 86.05% and 96.75% respectively. The results substantiate that the ZOC has excellent protective nature among the three different metal oxide impregnated coating used.

3.2.5.2. Potentio-dynamic polarization analysis

Potentio-dynamic polarization curve of NFCs and bare steel are recorded in 3.5 % NaCl aqueous solution. The obtained Tafel plot is shown in figure 12. A series of electrochemical measurements are carried out to evaluate
Figure 9. Contact angle measurements with water, isopropyl alcohol and ethylene glycol (a) TOC (b) ZOC (c) ZrOC.

Figure 10. Zisman plot showing surface tension NFCs (a) TOC (b) ZOC (c) ZrOC.
the corrosion potential ($E_{corr}$), corrosion current ($I_{corr}$) in the corrosion medium (acids) to justify the superior corrosion protecting performance of NFCs. Extrapolating the cathodic and anodic polarization curves to their point of intersection provides the $E_{corr}$ and $I_{corr}$ values.

In the case of TOC, the measured $E_{corr}$ value increased from $-625$ mV (bare steel) to $-474$ mV. The observed positive shift in the $E_{corr}$ value of about 151 mV and the decrease in $I_{corr}$ value (from $1.866 \times 10^{-3}$ A to $1.193 \times 10^{-4}$ A) is attributable to the corrosion inhibition property of coating. Similarly, the ZnO ($-0.456$ mV) and ZrO$_2$ ($-0.553$ mV) based NFCs has a positive shift of 169 mV and 72 mV, with a decrease in $I_{corr}$ value of $5.513 \times 10^{-5}$ A and $8.881 \times 10^{-5}$ A respectively. Uniformly dispersed metal oxide in an alkyd matrix acts as a strong barrier, inhibiting the penetration of corrosive ions towards the metal interface and thus protecting the metal from corrosion. The protection efficiency of the film is determined from the factors obtained from the Tafel plot as 93.61%, 97.05% and 95.24% for TOC, ZOC and ZrOC respectively.
3.2.6. NIR reflectance of NFCs

The NIR reflectance of NFCs was the most prominent features of the coating. The NFCs were coated on the glass substrate and their reflectance in the near infrared region was measured. The NIR reflectance of the coating was measured by NIR reflectance spectroscopy in the region of 750 nm to 2250 nm. Figure 13 shows the NIR reflectance of the uncoated and various NFCs on the glass. It is observed that for the TOC, the reflectance of around 40% to 58% in the heat producing infrared region of 750 nm to 1100 nm was observed. This high reflectance of 68% is achieved by the high surface area of the pigment present in the NFC. Those NPs having high refractive index reflect the heat producing near infrared radiation back into environment. Looking on to the NIR reflectance of ZOC, 40% to 68% of reflectance is observed in the region of 750 nm to 1100 nm. ZrOC shows the reflectance of 30 to 48% in the region of 750 nm to 1100 nm considered to be major heat influencing region in the infrared radiation. The ZOC was found to have high reflectance among other NPs dispersed coating. The higher reflectance of the near infrared region is due to the high refractive index property of the NPs which have
high surface area. Thus, the ZOC was optimized as the best nanocoating in accordance with the NIR and anticorrosion studies of NFCs.

Since from the NIR reflectance measurement of NFCs, it is found that about 40%–70% of heat producing section of near infrared light have been remarkably reflected. Making use of this reflecting property, the paint is coated over a prototype dwelling structure to study the temperature effects on the NFCs. To study these effects, a prototype made using glass in dimensions of length 6 inches, width 3 inches and height 6 inches is used. Initially the temperature measurement was done for the glass model by inserting the temperature sensing probe into the provision made at the bottom of the glass model of dimension of 0.5 inches diameter. Further, the study was carried out by ZOC over the fabricated model of glass house and the temperature change inside the model was examined.

The temperature change was studied for uncoated glass model, model coated with ZOC and glass model coated with commercial white paint. The glass model was kept in the sunlight around noon (11:00 am to 12:00 pm) for a period of one hour and the changes in the temperature were noted are given in the figures 14(a)–(c). The temperature of the uncoated glass was around 48 °C and that of the glass model coated with commercial paint was found to be 42.6 °C and the glass model coated with ZOC was about 40 °C. Among the three models, the one coated with ZOC shows a lower temperature inside, in contrast to the uncoated and the model coated with commercial paint maintain lower temperature inside.

4. Conclusion

In summary, NPs with high refractive index were synthesized by sol-gel, gel combustion and hydrothermal method. The synthesized NPs were characterized by XRD and FESEM. Efficient cool NFCs were prepared by high energy ball mill and the physical properties like glossiness, spreading area, hiding power and solid content of the paint were tested according to the ASTM standards. The FTIR analysis of the NFCs ensures the binding of the NPs to the resin. The NIR reflectance of the alkyd nanocoating was studied using NIR reflectance spectroscopy. Among the NFCs, the ZOC was found to show NIR reflectance of around 40 % to 68 % in the heat producing NIR region of 750 nm to 1100 nm. A prototype using glass was fabricated and coated with commercially available white paint and the ZOC. Upon keeping in sunlight around noon for 1 h, the interior temperature of the model was recorded. In contrast to the uncoated glass model and the commercially available white paint coated model, the ZOC maintains the interior temperature with a difference of 2.7 °C. Such a reduction into the interior temperature is a remarkable feature since it can minimize the costing load if the NFCs are used as coating for protecting structures. Also, the NFCs possess good aesthetic appearance, high corrosion inhibition property and possess good wetting ability, So ZOC in future can be used as a cool paint, which definitely minimizes the interior temperature to 2 °C–3 °C and thereby minimize the energy needed for cooling purposes.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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