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Development of Superhydrophobic Cotton Fabric Using Zinc Oxide Nanoflower/Polydimethylsiloxane (PDMS) Nanocomposite Coatings

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Article

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ABSTRACT

Nanoflower is anticipated to become a very smart material due to its unique properties such as high surface to volume ratio. A hydrothermal method was used in this study to prepare the zinc oxide (ZnO) nanoflower and characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FT-IR). The average particle size of the ZnO nanoflower was calculated as 21nm according to the Debye-Scherrer formula. The SEM result gives the surface morphological information of the ZnO nanoflower, which confirms the formation of the ZnO nanoflower. The ZnO nanoflower was dispersed in PDMS and coated onto cotton fabric to get the superhydrophobic fabric. The hydrophobicity was determined by measuring the water contact angle by the Sessile drop method and it was observed that coated fabrics have the highest contact angle, 140^o at 0.5% ZnO nanoflower concentration. The present study offers a method of fabrication of superhydrophobic cotton textile using ZnO nanoflower/PDMS polymer nanocomposites.

KEYWORDS

ZnO nanoflower, SEM, FT-IR, ZnO/PDMS, Superhydrophobicity

INTRODUCTION

There is intensive demand for multifunctional fabrics with different types of properties that have gained popularity and sparked the interest among industries and researchers. Different functional properties can be imparted to fabrics, making them fire retardant, water repellent (superhydrophobic), self-cleaning, UV absorbent, etc. [1,2]. The wetting capability of a solid surface is managed by its gradable surface topography and intrinsic chemical composition [3,4]. There are several approaches to meeting requirements for cellulose-based materials such as cotton to have superhydrophobic properties. Super hydrophobic textiles, because of their self-cleaning capabilities, have reduced the number of launderings. As the number of launderings is reduced, the functionalized textile's performance can be prolonged. In this regard, superhydrophobic fabric is eco-friendly, based on reduction in consumption of resources and energy required for laundering

[5-7]. The superhydrophobic surface-based textile materials have been fabricated from different inorganic materials and organic polymers [8].

Superhydrophobic textiles with appropriate surface roughness and suitable chemistry have been used for a wide range of applications, such as oil/water separation, self-cleaning, and multifunctional materials with the properties of UV-shielding, being flame-retardant, anti-icing, and photocatalytic properties, as well as materials for self-healing, responding to stimuli, patterning and asymmetric response [9-18].

Nanoflowers are a class of nanomaterials of research interest that can be synthesized by simple methods from organic and inorganic materials and also by the combination of both organic and inorganic materials with improved stability and efficiency of the surface reaction. Nanoflowers have several layers of petals to encompass a large surface area in a small nanostructure which can be used for many applications. The efficiency of the surface reaction has been increased by the 3D structure of nanoflowers. The number of adsorption sites is increased by the 3D structure of zinc oxide nanoflowers, which leads to increased efficiency of the zinc oxide nanoflowers [19]. ZnO nanostructures are biofriendly absorbers of UV radiation, nontoxic and chemically inert under exposure to high temperatures. Currently, the researchers have developed many different approaches to fabricating ZnO nanostructures. A zinc oxide nanoflower photocatalyst from the sea buckthorn fruit was synthesized for the degradation of industrial dyes in wastewater by E. J. Rupa et al. [20]. Microwave assisted fabrication of flower-like ZnO nanorods (NRs) for detecting reducing gasses was done by Abdullah et al. [21]. Hydrothermal growth of ZnO nanostructures is one of the most widely explored methods. The hydrothermal method can present good productivity without using any rigorous conditions or sophisticated instrumentation [22-24].

The hydrophobic organic polymers generally used in polymer nanocomposites are poly(tetrafluoroethylene) (PTEE), trimethoxy propyl silane (TMPSi), polyvinylidene fluoride (PVDF), polydimethylsiloxane (PDMS) [25-27]. The selection of a hydrophobic polymer is difficult because the degradation of some fluoropolymers is known to form perfluorooctanoic acids, which are very harmful to nature and human beings [23]. PDMS is one of the best organic polymers for the synthesis of the superhydrophobic surface because of its unique properties, such as hydrophobicity, excellent thermal stability, low glass transition temperature, good electrical properties, good weather resistance, low surface free energy, low toxicity and low chemical reactivity [29,30]. Both PDMS and ZnO nanoflower have exceptional non-toxic and environmentally friendly properties. Earlier ZnO/PDMS composites were synthesized by an electrodeposition-grafting modification method by P. T. Wang et al. [31]. PDMS composites filled with Tetrapodal ZnO-PDMS with ZnO fillers of different sizes and shapes were studied by Xin Jin et al. [32]. Sabino et al. developed a nanogenerator (NG) fabricated with composite with variable concentrations of NRs, grown by microwave radiation-assisted hydrothermal synthesis and PDMS. [33]. D. H. Kim et al. synthesized flower-like zinc oxide (ZnO) nanoarchitectures by a chemical precipitation method [34]. These nanoarchitectures were used to design an MWCNT (multiwalled carbon nanotube)/ZnO/PDMS composite film-based hybrid nanogenerator. In this study, the ZnO nanoflowers were used as the piezoelectric material as well as to increase the surface roughness of PDMS. In another study, a simple approach for maintaining superhydrophobic behaviour in the presence of oil contamination has been carried out. This is achieved through the use of a composite structure comprised of hydrophobized zinc oxide (ZnO) powder cast on a PDMS film. These composite surfaces are shown to be superhydrophobic as well as superoleophobic. [35]. A method to prepare zinc oxide (ZnO) nanoparticles and poly (methyl methacrylate) (PMMA) nanocomposites, which involved a covalent bonding through surface-initiated atom transfer radical polymerization (ATRP), was reported by Mojtaba et al. [36]. The above-mentioned

research work on ZnO/PDMS composites does not pertain to textile substrates. Earlier, one of the authors of this present work developed superhydrophobic polyamide fabrics using ZnO–PMMA nanocomposites [37]. In our present work, superhydrophobic textile surfaces have been fabricated by using ZnO/PDMS nanocomposites prepared by the solution mixing method. Most of the methods suggested in the literature involve complex steps and processes for the preparation of superhydrophobic surfaces. The novelty of the present work is that the ZnO/PDMS composites were prepared by simple solution mixing and applied to cotton fabrics by dip coating. This research work successfully leads to the creation of superhydrophobicity on cotton fabric with a low concentration of ZnO nanoflower in the composite. The materials used for the preparation of ZnO/PDMS are fluorine-free and commercially available on a large scale. The process can be easily incorporated into traditional textile industrial processes and it shows promise in view of the mass production of eco-friendly superhydrophobic fabrics.

EXPERIMENTAL

Materials and Methods

Zinc acetate (183.48 g/mol, 99.8%), sodium citrate (258.06 g/mol), sodium hydroxide (39.99 g/mol), PDMS and the curing agent SYLGARD® 184 (vinyl-terminated PDMS) were purchased from Sigma Aldrich. All chemicals were AR grade and used as received. A fine medium-weight 100%-white cotton fabric of plain weave was purchased from the local market. The fabric was washed with a detergent and dried in air before use. The chemical composition of the synthesized ZnO nanoflower and nanocomposites was examined by FT-IR spectroscopy with Nicolet is50 spectrometer in the wavelength range of 4000-400 cm^{-1}. For the FT-IR characterisation, powdered samples of the ZnO nanoflower, and polymer nanocomposites were used. The grain size of the ZnO nanoflower and the X-ray diffraction pattern were analysed by the d8 X-ray diffractometer using cu kα radiation at the wavelength λ=1.54 å. The structural parameter of the synthesized ZnO was obtained by the Rietveld refinement of the XRD pattern, using full professional software. The surface morphology (shape and size) and elemental composition of the ZnO nanoflower and the ZnO/PDMS composite were determined by SEM, instrument model JCM-6000PLUS (acc. voltage: 15.0 kV, probe current: 7.47500 nA, PHA mode: T3, Real Time: 30.13 sec). The superhydrophobicity of the polymer nanocomposite (ZnO/PDMS)-coated textile was determined by the Sessile drop method using Krüss Advance 1.6.2.0 instrument at IIT Delhi. The coated textile samples were cut into 15 mm (0.5”). The thickness of the sample was about 0.5 mm. In the present study, twenty different points on each sample were considered.

ZnO Nanoflower Synthesis

The ZnO nanoflower was synthesized by the hydrothermal method, which is simple and effective. 0.549 gm of zinc acetate and 0.735 gm of sodium citrate were dissolved in 40 ml of distilled water under constant stirring till a clear solution was obtained. Then 16 gm of sodium hydroxide was dissolved in 100 ml of distilled water under vigorous stirring and 4 ml of the NaOH solution was added to the above prepared solution. Then the mixture was taken in a 50 ml volume Teflon autoclave and heated at 120 °C for 8 hrs on the hot plate. After cooling to room temperature, the white precipitate was separated from the solution by centrifugation and then the solution was filtered and washed with distilled water and absolute ethanol 2-3 times and dried at 60 °C for 2 hrs on the hot plate.

Coating Cotton Fabric with ZnO/PDMS Nanocomposite

PDMS was dissolved in toluene in order to prepare a stock solution. Different percentages of ZnO nanoflower (0.1, 0.5, 0.8 and 1) were mixed with PDMS. The mixture was sonicated for 15 min to get a homogenous dispersion. After that, the curing agent was added to the stock solution, and then degasification was done for one hour to remove air bubbles. The cotton textile (3x3 cm) was dipped in the above solution for 2 hrs. After that, the textile was air-dried. The add-on percentage is the weight of the composite solution that has been added onto the textile surface and controls the evenness and thickness of the sample. The add-on percentage on the coated cotton fabric was calculated by using the following formula

$$\text{Add-on (\%)} = [(Y - X)/X] \times 100 \text{----------} (1),$$

where X is the weight of the uncoated fabric and Y is the coated fabric, which was about 20%.
Figure 1 shows the schematic illustration of the preparation of the ZnO nanoflower and the fabric coated with ZnO/PDMS nanocomposite.

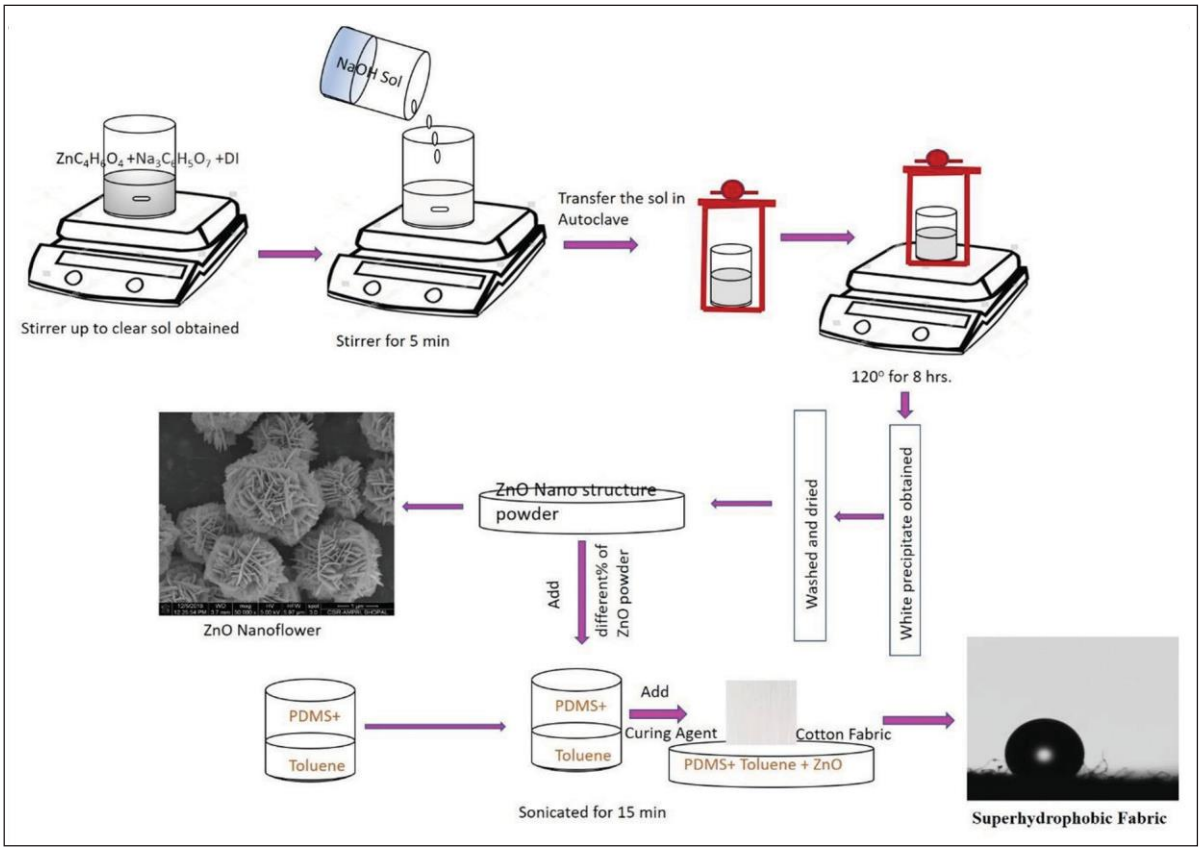

Figure 1. Schematic representation of the preparation of the ZnO nanoflower and the fabric coated with ZnO/PDMS nanocomposite

RESULTS AND DISCUSSION

Characterization of ZnO Nanoflower

XRD of ZnO Nanoflower

From Figure 2 it can be seen that all the peaks are corresponding to the wurtzite hexagonal structure given in the standard data file of JCPDS [38]. The XRD of the synthesized ZnO shows broad peaks values of 31.5, 34.2, 36.0 47.3 56.4, 62.7, 66.1, 66.7, 68.9, 72.4 and 89.4 which are typical for a ZnO structure. The peak

broadening in the XRD pattern indicates that a small nanocrystal is present in the samples [39,40]. The full-width half maxima (FWHM) of the diffraction peaks aided to determine the average crystalline (particle) size using the Debye-Scherrer equation (eq. 1). The average crystalline (particle) size of the ZnO nanoflower was 21nm. ZnO is crystallized in a hexagonal wurtzite structure with $P6_3mc$ space group. The lattice parameters of ZnO were determined as a=b=3.2498Å and c=47.5963Å.

$$D=K\lambda/\beta Cos\theta \text{---------- (2)},$$

where *D* is the crystalline size, *K* is the constant (for cu α K is 0.9), λ is X-ray wavelength, β is the full-width half maxima and θ is the Bragg angle.

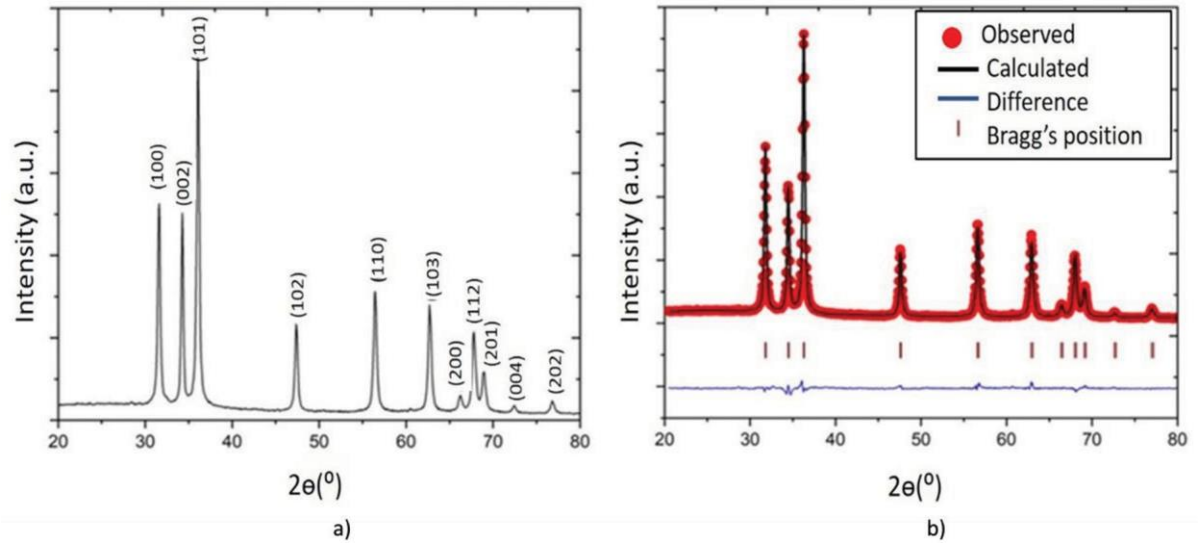

Figure 2**.** X-ray diffraction pattern of the ZnO Nanoflower

FTIR of ZnO Nanoflower

The FT-IR technique helped us analyse the possible interaction between the molecules or neighbouring structures in practical applications. The FT-IR spectra of the samples measured in the range of 4000-400 cm^{-1} are shown in Figure 3. The bands in this range are caused by ion vibrations in the crystal lattice. Metal oxides exhibit absorption bands in the fingerprint region, i.e. below 1000 cm^{-1}, due to inter-atomic vibrations. It is clear from the figure that the prepared ZnO peaking at 405 cm^{-1} and 855 cm^{-1} is due to the ZnO stretching and deformation vibration, respectively. The peak at 3280 cm^{-1} is due to the presence of O-H stretching vibration [41-44]. FTIR of PDMS exhibiting IR peaks at 2961 cm^{-1} is attributed due to the asymmetric CH_3 stretching in Si-CH_3, the peak at 1259 cm^{-1} is observed due to the CH_3 deformation in Si-CH_3, the one at 1011 cm^{-1} occurred due to Si-O-Si stretching and the one at 787 cm^{-1} is due to -CH_3 rocking and Si-C stretching in Si-CH_3 [45].

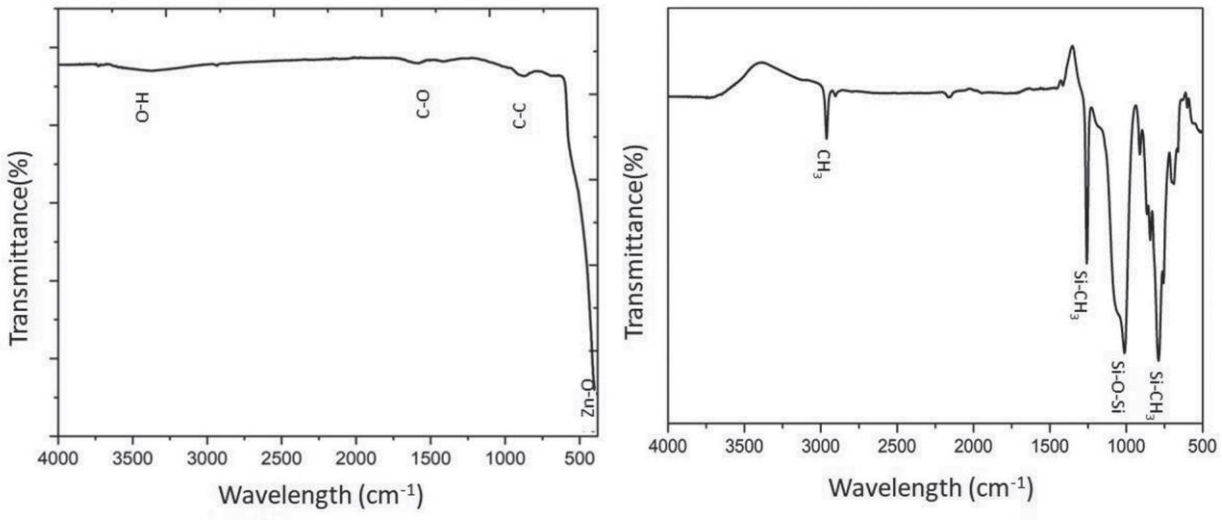

Figure 3. (a) FTIR of the ZnO Nanoflower Figure 3. (b) FTIR of polydimethylsiloxane (PDMS)

FESEM and EDX of ZnO Nanoflower

The surface structures and morphologies of the as-synthesized ZnO nanostructure were examined by SEM. Figure 4 (a) shows the image of the prepared ZnO nanostructure in a flower shape confirming the formation of a nanoflower. All the ZnO nanoflower samples were composed of a large number of nano-petals. The diameter of nano-petals comes in the range of 40-50 nm. Figure 4 (b) shows EDX analysis of the ZnO, which confirms the presence of Zn and O in the required ratio in the synthesized material. Figure 4 (c), the SEM image of PDMS, and Figure 4 (d), EDX of PDMS, show the presence of silicon (Si) peaks, which confirms that it is silicon-rich with the weight percentage of 66.54.

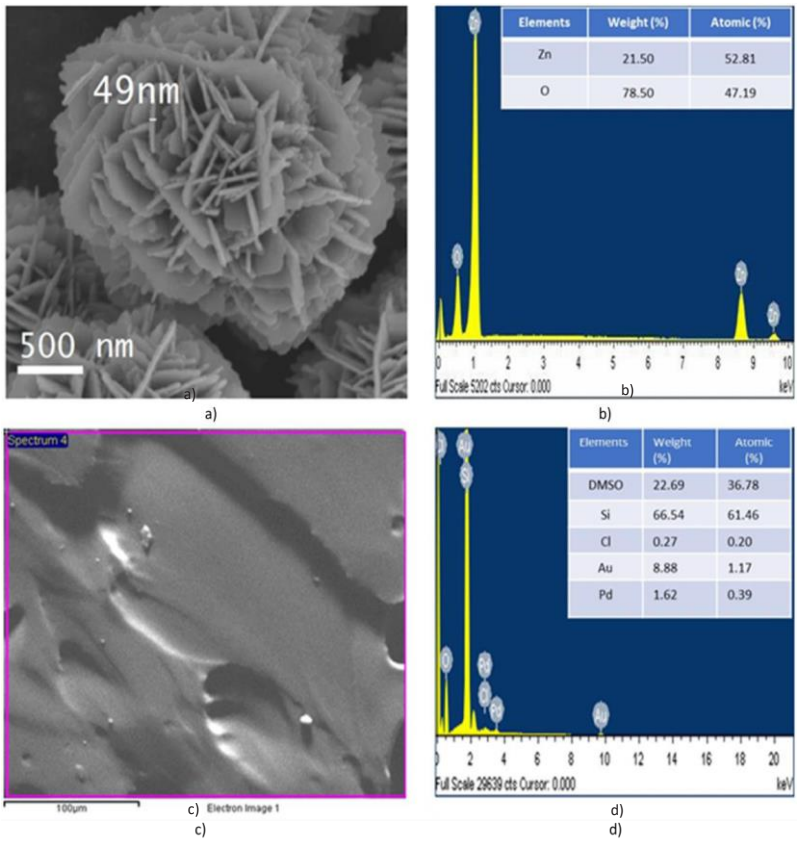

Figure 4. (a, b) FESEM and EDX of the ZnO Nanoflower and (c, d) FESEM and EDX of PDMS

Characterization of ZnO/PDMS Polymer Nanocomposite

FESEM of ZnO Nanoflower/PDMS

Figure 5 shows the SEM of different concentrations of ZnO nanoflower/PDMS. Figure 5 (a-e) shows that the polymer nanocomposite with different concentrations (0.1, 0.5, 0.8 and 1.0% of ZnO) exhibited a similar rough and porous microstructure with randomly distributed micron-size bumps. The bumps are due to the aggregate of the ZnO nanoflower. The direct mutual attraction between nanoparticles via van der Waals forces or chemical bonds causes aggregation/agglomeration of nanoparticles. The higher magnification in Figure 5 (d) with 0.5% of ZnO shows the porous structure with spherical nanoparticles of about 100-600 nm. Figure 5 (e) is SEM of 1%-concentrations of ZnO nanoflower/PDMS.

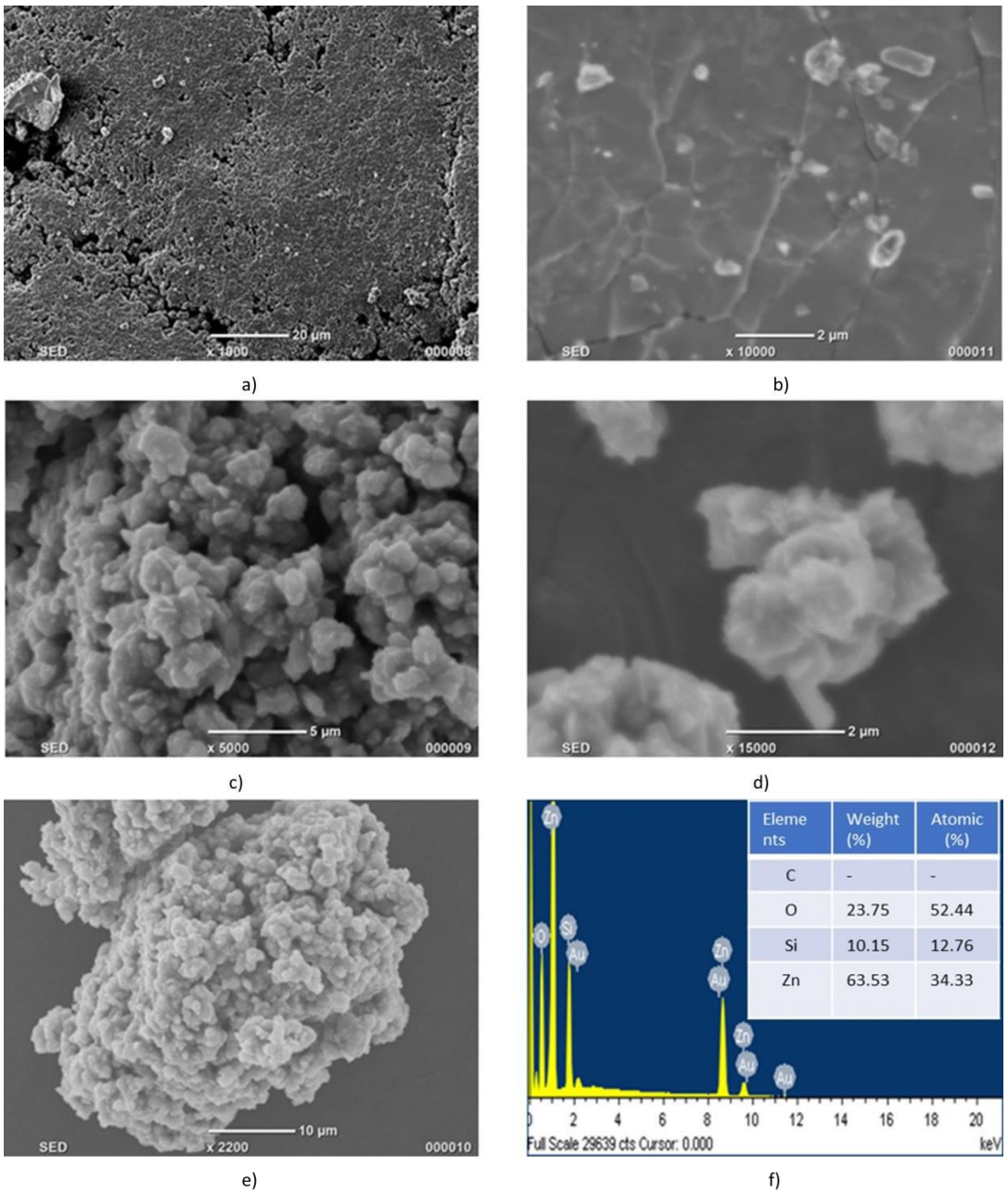

Figure 5. FESEM and EDX of the ZnO nanoflower/PDMS nanocomposites. (a) FESEM of 0.1% of the ZnO nanoflower/ PDMS nanocomposites; (b) FESEM of 0.5% of the ZnO Nnnoflower/PDMS nanocomposites; (c) FESEM of 0.8% of the ZnO nanoflower/PDMS nanocomposites; (d) Higher magnification of 0.5% of the ZnO nanoflower/PDMS nanocomposites; (e) FESEM of 1.0% of the ZnO nanoflower/PDMS nanocomposites; (f) EDX of the ZnO nanoflower/PDMS nanocomposites

Figure 5 (f), EDX of ZnO/PDMS polymer nanocomposites, shows that the composite is composed of elements of carbon, oxygen, silicon, and zinc. Among them, silicon and zinc are accounting for 8.12% and 64.94%, respectively. Silicon was derived from PDMS, and zinc from ZnO.

FTIR of ZnO Nanoflower/PDMS

The FTIR spectrum of different concentrations of the ZnO nanoflower/PDMS superhydrophobic coating is shown in Figure 6. The band at 2961 cm^{-1} is attributed to the O-H stretching band vibration. It was due to a small amount of water being adsorbed on the ZnO surface [46]. The peak that occurred at 1258 cm^{-1} was due to the $–CH_3$ group. The band at 1065 and 1012 cm^{-1} was due to the stretching modes of Si-O-Si bonds. A small peak at 863 cm^{-1} observed in ZnO/PDMS could be originating from the Zn-O-Si bonding. The peaks at 863 and 843 cm^{-1} show weak ZnO absorption. The bands at 791 and 756 cm^{-1} were due to the symmetric Si-O stretching mode. The peaks at 417 and 405 cm^{-1} corresponded with the ZnO stretching vibration. This observation gives an idea that the ZnO nanoflower reacts also with some residual silanol (Si-OH) groups. The presence of methyl groups contributes to the superhydrophobic properties and, from these spectra, it can be concluded that the PDMS/ZnO composites have methyl group, which improves the hydrophobic behaviour. The overlapping of peaks of polymer composites with different concentrations of the ZnO nanoflower shows that the chemical composition remains the same with the different concentrations of the ZnO nanoflower.

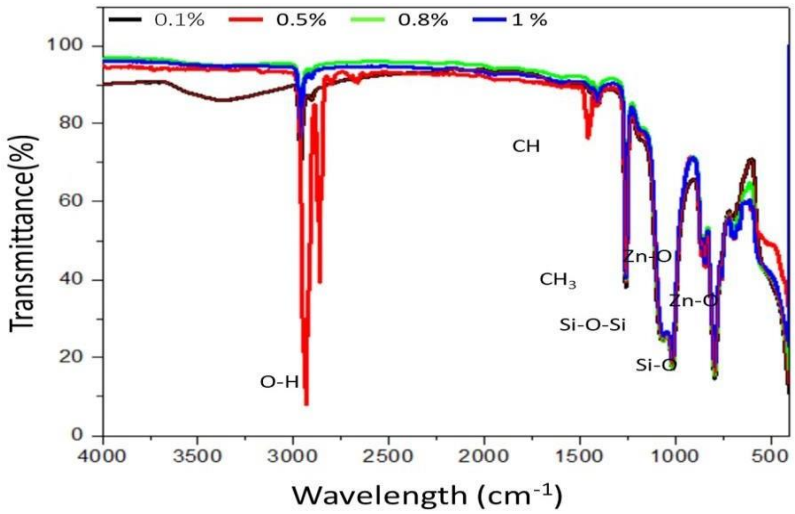

Figure 6**.** FTIR of the ZnO nanoflower/PDMS nanocomposites (different percentages of ZnO)

Measurement of Superhydrophobicity of Coated Textile

The contact angle of the PDMS-treated fabric as reported in the literature is around 130^o [47,48]. It is known, however, that a pure PDMS coating using conventional coating methods will not lead to a superhydrophobic surface [49]. In the present study, twenty different points on each sample were considered.

The contact angle of the textile coated with ZnO/PDMS with different ZnO nanoflower concentrations is shown in Figure 7. Contact angles are 131^o, 140^o, 138^o and 135^o with 0.1%, 0.5%, 0.8% and 1.0% concentrations. At 0.5 % of the ZnO nanoflower concentration the maximum contact angle of 140° was obtained here due to the formation of micro-nano roughness by ZnO nanoflower nanoparticles as well as the low surface energy material, PDMS.

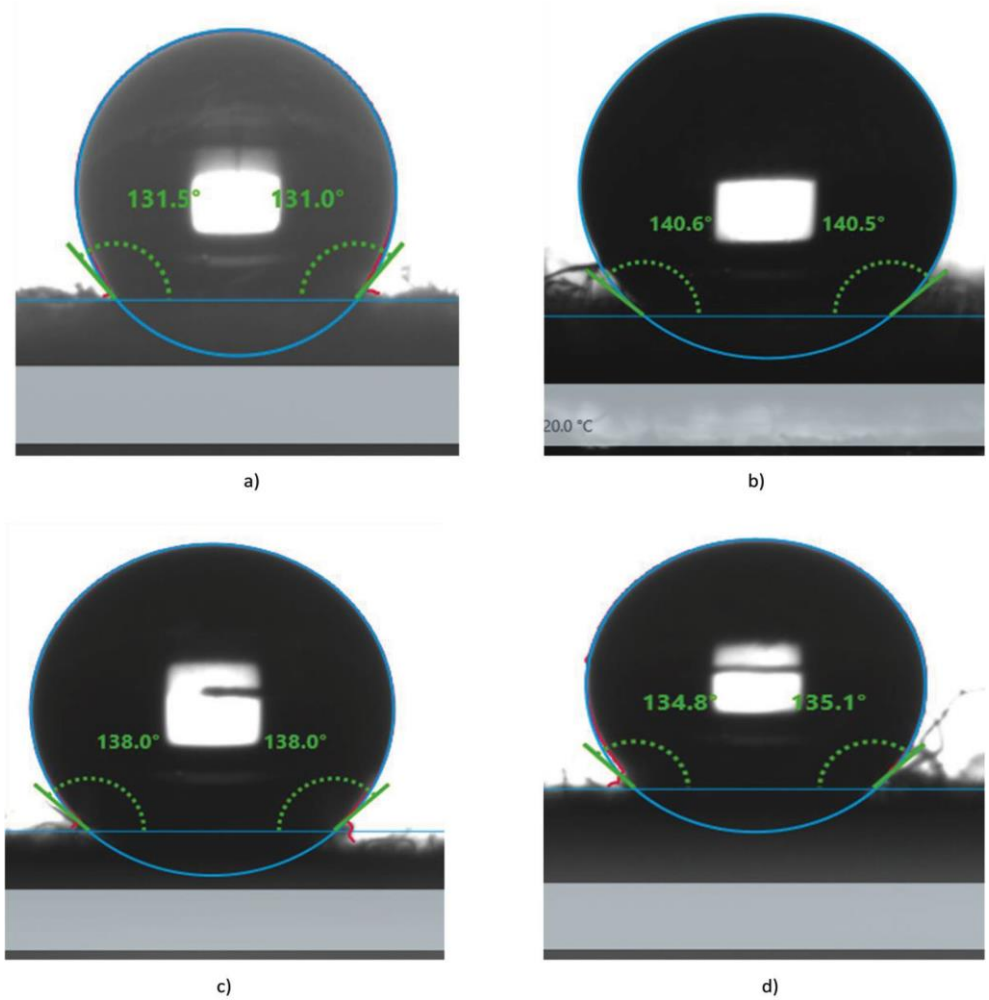

Figure 7. Contact angle of the ZnO nanoflower/PDMS nanocomposites: (a) Contact angle - 131° at 0.1%; (b) Contact angle -140°at 0.5%; (c) Contact angle - 138° at 0.8%; (d) Contact angle - 135° at 1.0%

The relationship between the mean contact angle (with standard deviation) and the concentration percentage of the ZnO nanoflower powder is illustrated in Figure 8. The figure illustrates that with an increase of the ZnO nanoflower concentration, the contact angle increases until a certain concentration level. After that, with a further increase in concentration, the contact angle decreases. It was found that superhydrophobicity can be achieved at the ZnO concentration of 0.5%. Further increment in concentration does not improve the superhydrophobicity. The reason could be the particle size or their nature has more effect at the minimum particle concentration that is essential to attain superhydrophobicity. From the water contact angle analysis, it is observed that once superhydrophobicity is achieved, a further increase in the concentration of the nanoflower in the composite does not have any significant effect on the wettability of the textile surfaces.

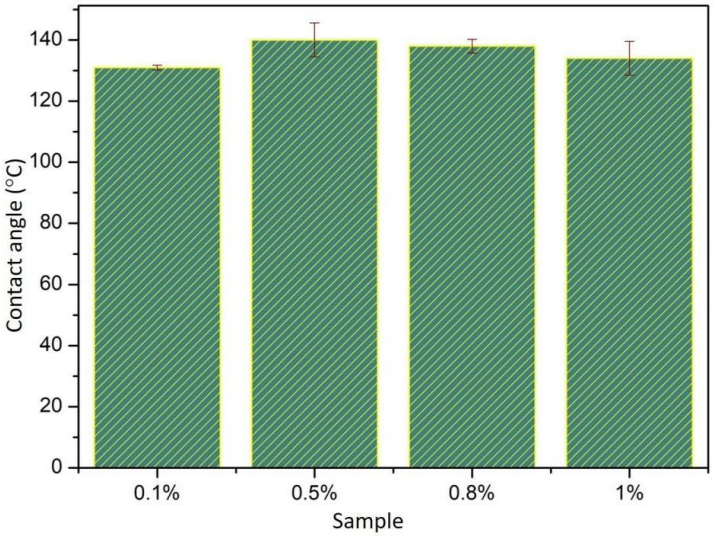

Figure 8. Contact angle versus percentage of the ZnO nanoflower in the ZnO/PDMS nanocomposite

CONCLUSION

The fabrication of a superhydrophobic surface using ZnO nanoflowers prepared by hydrothermal process and subsequently making composite with PDMS with a simple solution mixing method was demonstrated. The ZnO nanoflower was prepared using the hydrothermal method and analysed through X-ray diffraction, FESEM and FTIR. The average grain size of a ZnO nanoflower was calculated to be 21 nm according to the Debye-Scherrer formula. The nanoflower exhibits a hexagonal wurtzite structure with a particle size of 21 nm. FESEM result provides information on the morphology of the nanoparticle, which is in a flower-like shape. The FESEM of the polymer nanocomposite shows rough and porous microstructure with micron-size bumps. The FTIR analysis of ZnO/PDMS confirms that ZnO is chemically bonded to PDMS. The combination of PDMS and the ZnO nanoflower in a nanocomposite coating on textile results in superhydrophobicity. ZnO at a concentration of 0.5% in the polymer nanocomposite leads to superhydrophobicity in the textile. The advantages of the present method include the use of cheap and fluorine-free raw materials, avoiding hazardous solvents and the possibility for application on a large area of various substrates.

Author Contributions

Conceptualization – Vijaya Kumar SG, Srivastava AK; methodology – Vijaya Kumar SG; formal analysis – Vijaya Kumar SG, Khan MA; experiments – Prabhakar P, Sen RK, Uppal N; writing-original draft preparation – Vijaya Kumar SG, Prabhakar P; writing-review and editing – Khan MA, Srivastava AK; supervision – Vijaya Kumar SG. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

REFERENCES

[1] Zhao Q, Wu LYL, Huang H, Liu Y. Ambient-curable superhydrophobic fabric coating prepared by water-based non-fluorinated formulation. Materials & Design. 2016; 92:541–545. http://dx.doi.org/10.1016%2Fj.matdes.2015.12.054

[2] Aadhar AM, Patel PN, Satyajeet BC, Bharat HP. Preparation of flame-retardant polymer nanocomposite textiles. Chemical Fibers International. 2016; 66(4):190-191. https://www.researchgate.net/publication/312212896

[3] Latthe SS, Demirel AL. Polystyrene/octadecyltrichlorosilane superhydrophobic coatings with hierarchical morphology. Polymer Chemistry. 2013; 4(2):246–249. https://doi.org/10.1039/C2PY20731A

[4] Simpson JT, Hunter SR, Aytug T. Superhydrophobic materials and coatings: a review. Reports on Progress in Physics. 2015; 78(8):086501. https://doi.org/10.1088/0034-4885/78/8/086501

[5] Sethi SK, Manik G. Recent Progress in Super Hydrophobic/Hydrophilic Self-Cleaning Surfaces for Various Industrial Applications. A Review. Polymer-Plastics Technology and Engineering. 2018; 57(18):1932-1952. https://doi.org/10.1080/03602559.2018.1447128

[6] Park S, Kim J, Park CH. Superhydrophobic Textiles: Review of Theoretical Definitions, Fabrication and Functional Evaluation. Journal of Engineered Fibers and Fabrics. 2015; 10(4):1-18. https://journals.sagepub.com/doi/pdf/10.1177/155892501501000401

[7] Barthlott W, Barthlott CN. Purity of the sacred lotus a escape from contamination in biological surface. Planta. 1997; 202:1–8. https://doi.org/10.1007/s004250050096

[8] Hoefnagels HF, Wu D, de With G, Ming W. Biomimetic Superhydrophobic and Highly Oleophobic Cotton Textiles. Langmuir. 2007; 23(26):13158–13163. https://doi.org/10.1021/la702174x

[9] Cao LL, Jones AK, Sikka VK, Wu JZ, Gao D. Anti-icing superhydrophobic coatings. Langmuir. 2009; 25(21):12444-8. https://doi.org/10.1021/la9028

[10] Wang HX, Zhou H, Gestos A, Fang J, Niu HT, Ding J,Lin T. Robust, electro-conductive, self-healing superamphiphobic fabric prepared by one-step vapour-phase polymerisation of poly(3, 4-ethylenedioxythiophene) in the presence of fluorinated decyl polyhedral oligomeric silsesquioxane and fluorinated alkyl silane. Soft Matter. 2013; 9:277-282. https://doi.org/10.1039/C2SM26871J

[11] Arslan O, Aytac Z, Uyar T. Superhydrophobic, hybrid, electrospun cellulose acetate nanofibrous mats for oil/water separation by tailored surface modification. ACS Appl. Mater. Interfaces. 2016; 8(30):19747-54. https://doi.org/10.1021/acsami.6b05429

[12] Peng HL, Wang H, Wu JN, Meng GH, Wang YX, Shi YL, Liu ZY, Guo XH. Preparation of superhydrophobic magnetic cellulose sponge for removing oil from water. Ind. Eng. Chem. Res. 2016; 55(3):832-838. https://doi.org/10.1021/acs.iecr.5b03862

[13] Shibraen MHMA, Yagoub H, Zhang XJ, Xu J, Yang SG. Anti-fogging and anti-frosting behaviors of layer-by-layer assembled cellulose derivative thin film. Applied Surface Science. 2016; 370:1-5. https://doi.org/10.1016/j.apsusc.2016.02.060

[14] Jiang B, Zhang HJ, Zhang LH, Sun YL, Xu LD, Sun ZN, Gu WH, Chen ZX, Yang HW. Novel one-step, in situ thermal polymerization fabrication of robust superhydrophobic mesh for efficient oil/water separation. Engineering Chemistry Research. 2017; 56(41):11817–11826. https://doi.org/10.1021/acs.iecr.7b03063

[15] Chen SS, Li X, Li Y, Sun JQ. Intumescent flame-retardant and self-healing superhydrophobic coatings on cotton fabric. ACS Nano. 2015; 9(4):4070-4076. https://doi.org/10.1021/acsnano.5b00121

[16] Gao JF, Huang XW, Xue HG, Tang LC, Li RKY. Facile preparation of hybrid microspheres for super-hydrophobic coating and oil-water separation. Chemical Engineering Journal. 2017; 326:443-453. https://doi.org/10.1016/j.cej.2017.05.175

[17] Mi NY, XJing X, Huang HX, Peng XF, Turng L.S. Superhydrophobic graphene/cellulose/silica aerogel with hierarchical structure as superabsorbers for high efficiency selective oil absorption and recovery. Industrial & Engineering Chemistry Research. 2018; 57:1745-1755. https://doi.org/10.1021/acs.iecr.7b04388

[18] Schlaich C, Li MJ, Cheng C, Donskyi IS, Yu LX, Song G, Osorio E, Wei Q, Haag R. Mussel-inspired polymer-based universal spray coating for surface modification: fast fabrication of antibacterial and superhydrophobic surface coatings. Advanced Materials Interfaces. 2018; 5(5):1701254. https://doi.org/10.1002/admi.201701254

[19] Zhang G, Deng C, Shi H, et al. ZnO/Ag composite nanoflowers as substrates for surface-enhanced Raman scattering. Applied Surface Science. 2017; 402:154-160. http://dx.doi.org/10.1016%2Fj.apsusc.2017.01.042

[20] Esrat J R, Lalitha K, Suleman A, Deok-Chun Y, Seok-Kyu J. Synthesis of a Zinc Oxide Nanoflower Photocatalyst from Sea Buckthorn Fruit for Degradation of Industrial Dyes in Wastewater Treatment. Nanomaterials. 2019; 9(12):1692. https://doi.org/10.3390/nano9121692

[21] Abdullah A, Faheem A, Chawki A, Nagih MS. ′Flower-Like ZnO Nanorods Synthesized by Microwave-Assisted One-Pot Method for Detecting Reducing Gases: Structural Properties and Sensing Reversibility. Front Chemistry. 2020. https://doi.org/10.3389/fchem.2020.00456

[22] Kamegawa T, Irikawa K, Yamashita H. Multifunctional surface designed by nanocomposite coating of polytetrafluoroethylene and TiO2 photocatalyst: self-cleaning and superhydrophobicity. Scientific Reports. 2017; 7:13628. https://doi.org/10.1038/s41598-017-14058-9

[23] Ramanathan R, Weibel DE. Novel liquid–solid adhesion superhydrophobic surface fabricated using titanium dioxide and trimethoxypropyl silane. Applied Surface Science. 2012; 258(20):7950–7955. https://doi.org/10.1016/j.apsusc.2012.04.140

[24] Ganesh VA, Nair AS, Raut HK, Yuan Tan TT, He C, Ramakrishna S, Xu J. Superhydrophobic fluorinated POSS–PVDF-HFP nanocomposite coating on glass by electrospinning. Journal of Materials Chemistry. 2012; 22(35):18479-18485. https://doi.org/10.1039/C2JM33088A

[25] Kowalczyk D, Brzezinski S, Kaminska I. Multifunctional nanocoating finishing of polyester/cotton woven fabric by the sol-gel method. Textile Research Journal. 2017; 88(8):946–956. https://doi.org/10.1177/0040517517693979

[26] Ogihara H, Xie J, Okagaki J, Saji T. Simple Method for Preparing Superhydrophobic Paper: Spray-Deposited Hydrophobic Silica Nanoparticle Coatings Exhibit High Water-Repellency and Transparency. Langmuir. 2012; 28(10):4605–4608. https://doi.org/10.1021/la204492q

[27] Kathirvelua S, Louis D, Bharathi D. UV protection finishing of textiles using ZnO nanoparticles. Indian Journal of Fibre & Textile Research. 2009; 34(3):267-273. http://hdl.handle.net/123456789/6078

[28] Ganesh VA, Nair AS, Raut HK, Yuan Tan TT, He C, Ramakrishna S, Xu J. Superhydrophobic fluorinated POSS–PVDF-HFP nanocomposite coating on glass by electrospinning. Journal of Materials Chemistry. 2012; 22(35):18479-18485. https://doi.org/10.1039/C2JM33088A

[29] Zhang J, Pu G, Severtson SJ. Fabrication of Zinc Oxide/Polydimethylsiloxane Composite Surfaces Demonstrating Oil-Fouling-Resistant Superhydrophobicity. ACS Applied Materials & Interfaces. 2010; 2(10):2880–2883. https://doi.org/10.1021/am100555r

[30] Tinglan W, Zicheng L, Xueqi W, Zhicheng Z, Qi Z, Bing Y. A compound of ZnO/PDMS with photocatalytic, self-cleaning and antibacterial properties prepared via a two-step method. Applied Surface Science. 2021; 550:149286. http://dx.doi.org/10.1016/j.apsusc.2021.149286

[31] Mao D, Sören K, Xinwei Z, Iris H, Kristin M, Rainer A, Yogendra K M. Study of Tetrapodal ZnO-PDMS Composites: A Comparison of Fillers Shapes in Stiffness and Hydrophobicity Improvements. PLOS ONE. 2014; 9(9):e106991. https://doi.org/10.1371/journal.pone.0106991

[32] Andreia DS, Filipe S, Ana R, Pedro B, Hugo Á, Elvira F, Rodrigo M, Rui I. Optimization of ZnO Nanorods Concentration in a Micro-Structured Polymeric Composite for Nanogenerators. Chemosensors. 2021; 9:27. https://doi.org/10.3390/ chemosensors9020027

[33] Kim DH, Dudem B, Yu JS. High-Performance Flexible Piezoelectric-Assisted Triboelectric Hybrid Nanogenerator via Polydimethylsiloxane-Encapsulated Nanoflower-like ZnO Composite Films for Scavenging Energy from Daily Human Activities. ACS Sustainable Chemistry and Engineering. 2018; 6(7):8525-8535. https://doi.org/10.1021/acssuschemeng.8b00834

[34] Zhang J, Pu G, Severtson SJ. Fabrication of Zinc Oxide/Polydimethylsiloxane Composite Surfaces Demonstrating Oil-Fouling-Resistant Superhydrophobicity. ACS Applied Materials & Interfaces. 2010; 2(10):2880–2883. https://doi.org/10.1021/am100555r

[35] Mojtaba A, Nafiseh KA, Solmaz ES. Synthesis of Poly (methylmethacrylate)/Zinc Oxide Nanocomposite with Core-Shell Morphology by Atom Transfer Radical Polymerization. Journal ofMacromolecular Science. Part A: Pure and Applied Chemistry. 2013; 50:9: 966-975. https://doi.org/10.1080/10601325 .2013.813814

[36] Gowri SV, Almeida L, Teresa MP, Noemia C, Souto AP, Esteves MF, Sanghi SK. Functional finishing of polyamide fabrics using ZnO–PMMA nanocomposites. Journal of Materials Science. 2010; 45(9):2427–2435. https://doi.org/10.1007/s10853-010-4210-4

[37] Asthana A, Maitra T, Büchel R, Tiwari MK, Poulikakos D. Multifunctional Superhydrophobic Polymer/ Carbon Nanocomposites: Graphene, Carbon Nanotubes, or Carbon Black? ACS Applied Materials & Interfaces. 2014; 6(11):8859–8867. https://doi.org/10.1021/am501649w

[38] Powder diffraction file, alphabetical index inorganic phases: JCPDS International Centre For Diffraction Data 1601, 1984; Park Lane Swarthmore, Pennsylvania 19081 USA set 6–10:87.

[39] Fan J, LI T, Heng H. Hydrothermal growth of ZnO nanoflowers and their photocatalyst application. Bulletin of Materials Science. 2016; 39(1):19–26. https://doi.org/10.1007/s12034-015-1145-z

[40] Nagarajan S, Arumugam KK. Extracellular synthesis of zinc oxide nanoparticle using seaweeds of gulf of Mannar, India. Journal of Nanobiotechnology. 2013; 11(1):39. https://doi.org/10.1186/1477-3155-11-39

[41] Paino IMM, Gonçalves FJ, Souza FL, Zucolotto V. Zinc Oxide Flower-Like Nanostructures That Exhibit Enhanced Toxicology Effects in Cancer Cells. CS Applied Materials & Interfaces. 2016; 8(48):32699–32705. https://doi.org/10.1021/acsami.6b11950

[42] Haider AJ, Sultan FI, Al-Nafiey A, editors, Controlled growth of different shapes for ZnO by hydrothermal technique. In: AIP Conference Proceedings AIP Conference Proceedings 1968, 030085; 1-3 Feb 2018; Beirut, Lebanon. 2018. https://doi.org/10.1063/1.5039272

[43] Mohan S, Vellakkat M, Aravind A, Reka U. Hydrothermal synthesis and characterization of Zinc Oxide nanoparticles of various shapes under different reaction conditions Nano Express 2020; 1:030028. https://doi.org/10.1088/2632-959X/abc813

[44] Johnson M, Gao L, Shields W, Smithi M, Efimenko K, Cushing K, Genzer J and Lopez G. Elastomeric Microparticles for Acoustic Mediated Bioseparations. Journal of Nanotechnology 2013; 11:23. https:// doi:10.1186/1477-3155-11-22

[45] Chakradhar R, Kumar VD, Rao J, Basu BJ. Fabrication of superhydrophobic surfaces based on ZnO–PDMS nanocomposite coatings and study of its wetting behaviour. Applied Surface Science 2011; 257:8569-8575 https://doi.org/10.1016/j.apsusc.2011.05.016

[46] Ronggang C, Karine G, David DS, Myriam V, Nicolas M, Benoît K, Bernard N, Alain MJ. Environmentally friendly super-water-repellent fabrics prepared from water-based suspensions. ACS Applied Materials Interfaces. 2018; 10:15346−15351 https://doi.org/10.1021/acsami.8b02707

[47] Esfandiar P, Hai Z, Bin T, Russell V, Xungai W. Superhydrophobic and photocatalytic self-cleaning on cotton fabric using fluorine-free flower-like-TiO2/PDMS coatings. Research Square. https://doi.org/10.21203/rs.3.rs-404885/v1

[48] Wang E, Kashi J, Sun SL, Wang X. Advances in photocatalytic self-cleaning, superhydrophobic and electromagnetic interference shielding textile treatments. Advances in Colloid and Interface Science. 2020; 277:102116. https://doi.org/10.1016/j.cis.2020.102116