DEVELOPMENT OF SOLID-STATE FLUORESCENT CARBON DOTS AND THEIR APPLICATION TOWARDS SUPERHYDROPHOBIC COATINGS

M.Tech. Thesis

By

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DISCIPLINE OF METALLURGICAL ENGINEERING AND MATERIALS SCIENCE

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DEVELOPMENT OF SOLID-STATE FLUORESCENT CARBON DOTS AND THEIR APPLICATION TOWARDS SUPERHYDROPHOBIC COATINGS

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PRAVEEN RAUTELA

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INDIAN INSTITUTE OF TECHNOLOGY INDORE

CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **DEVELOPMENT OF SOLID-STATE FLUORESCENT CARBON DOTS AND THEIR APPLICATION TOWARDS SUPERHFROPHOBIC COATINGS** in the partial fulfillment of the requirements for the award of the degree of **MASTER OF TECHNOLOGY** and submitted in the **DISCIPLINE OF METALLURGICAL ENGINEERING AND MATERIALS SCIENCE, Indian Institute of Technology Indore**, is an authentic record of my own work carried out during the time period from August 2021 to May 2023 under the supervision Dr. Mrigendra Dubey, Associate professor, IIT Indore.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

05/06/23

Signature of the student with date
(PRAVEEN RAUTELA)

This is to certify that the above statement made by the candidate is correct to the best of my/our knowledge.

M. Dubey 05/06/23

Signature of the Supervisor of
M.Tech. thesis (with date)
(Dr. MRIGENDRA DUBEY)

PRAVEEN RAUTELA has successfully given his/her M.Tech. Oral Examination held on **25/05/2023.**

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Date:

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DEDICATION

Dedicated to my Guide

My Parents

My Teachers

My Seniors

My Friends

Abstract

This research work presents the synthesis of fluorescent hydrophobic carbon dots (CDs) using a solvothermal process and explores their applications in superhydrophobic coatings and fingerprint detection. The CDs were prepared by subjecting organic precursors to a solvothermal reaction, resulting in the formation of carbon dots with hydrophobic properties and strong fluorescence emission.

The solvothermal synthesis route provided a convenient and efficient method for producing hydrophobic CDs. The reaction parameters, such as temperature and time, were optimized to achieve desired fluorescence properties and hydrophobicity. The synthesized CDs exhibited excellent fluorescence characteristics, enabling their utilization in various optical applications.

The hydrophobic nature of the CDs facilitated their incorporation into superhydrophobic coatings. The CDs were uniformly dispersed within the coating matrix, contributing fluorescence properties without compromising the superhydrophobicity of the coatings. The resulting composite coatings displayed outstanding water-repellent properties and retained the fluorescence emission of the CDs. This outcome demonstrated the potential for developing functional coatings with enhanced optical and surface properties.

In addition to superhydrophobic coatings, the synthesized hydrophobic CDs were employed in fingerprint detection. By applying the CDs onto different substrates, including paper and glass, the inherent fluorescence of the CDs allowed for the visualization and enhancement of fingerprint patterns. The CDs selectively adhered to the ridges of the fingerprints, leading to improved contrast and clarity of the fingerprint details. This approach provided a straightforward and effective method for fingerprint detection without the need for complex chemical treatments.

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ACRONYMS

HCD – Hydrophobic carbon dots

AIE – Aggregation Induced Emission

FTIR – Fourier Transform Infrared

PVDF – Polyvinylidene Fluoride

Chapter 1

Introduction

1.1 Overview

The present report outlines the process of synthesising nanomaterials with dual properties of superhydrophobicity and fluorescence, utilising the solvothermal technique. The solvothermal technique is a highly effective method that enables accurate manipulation of the size and morphology of nanomaterials. Through the optimization of reaction parameters, such as temperature and pressure, researchers are able to customise the properties of nanomaterials to suit their particular requirements.

The important characteristic of the nanomaterials that have been developed is their superhydrophobic quality, which is integral to their potential applications. The process facilitates the creation of a hydrophobic coating on the nanomaterials' exterior, thereby safeguarding them against deterioration and erosion. This attribute renders them exceedingly sought-after for a diverse array of applications, encompassing sensors, coatings, and surfaces that are self-cleaning.

Moreover, the fluorescence characteristic of the synthesized nanomaterials confers upon them distinct sensing and imaging capabilities. Quantum dots are capable of emitting light upon excitation by an external light source, rendering them highly suitable for the purpose of detecting and monitoring alterations in the environment. This particular attribute presents novel prospects for their application in diverse domains, including but not limited to bioimaging, diagnostics, and environmental surveillance.

1.2 Carbon dots (nanomaterial)

Carbon dots are a type of nanomaterial that is based on carbon and is currently receiving growing attention in both research and technological applications. These are tiny fluorescent nanoparticles made up of carbon atoms with a diameter of less than 10 nm. Carbon dots are commonly produced using affordable and non-hazardous starting materials, such as citric acid, glucose, or ethylene glycol. Additionally, these dots can be adjusted with functional groups to regulate their optical and physical characteristics.

The optical properties of carbon dots are considered highly promising. These materials demonstrate vivid and adjustable luminescence throughout the visible and near-infrared portions of the electromagnetic spectrum, rendering them appealing contenders for deployment in applications related to imaging and sensing. Moreover, carbon dots exhibit minimal toxicity, favourable biocompatibility, and exceptional photostability, rendering them a highly coveted option for employment in biological contexts.

Carbon dots can be produced through diverse techniques, including solvothermal synthesis, hydrothermal synthesis, or microwave-assisted synthesis. Typically, carbon dots are produced through the process of carbonization of precursors under conditions of elevated temperature and pressure, which is subsequently followed by functionalization in order to confer particular properties.

Recent studies have exhibited the prospective utilization of carbon dots in diverse domains such as biological imaging, sensing, drug delivery, energy storage, and catalysis. Carbon dots have demonstrated remarkable imaging capabilities for both in vitro and in vivo biomedical applications, owing to their high biocompatibility and low toxicity. Furthermore, carbon dots have been explored as potential candidates for

drug delivery systems due to their diminutive dimensions, which facilitate effective cellular incorporation and precise drug transportation.

1.3 Superhydrophobicity

Superhydrophobicity is a remarkable phenomenon characterized by a surface's ability to display an exceptional degree of water repellency. This implies that water droplets are unable to easily wet or adhere to the surface but rather tend to roll off or rebound. The observed behavior can be attributed to the distinctive integration of surface chemistry and micro/nano-scale surface texture.

The origin of the term "superhydrophobic" can be traced back to its Greek roots, where "super" denotes a state of being above or beyond, and "hydrophobic" refers to the property of repelling water. A surface exhibiting superhydrophobicity is characterized by a contact angle with water that exceeds 150 degrees and a sliding angle that is typically less than 10 degrees. The contact angle refers to the geometric angle formed between the interface of a solid surface and the tangent to a liquid droplet at the point of contact. Conversely, the sliding angle is at which a droplet initiates sliding off the surface.

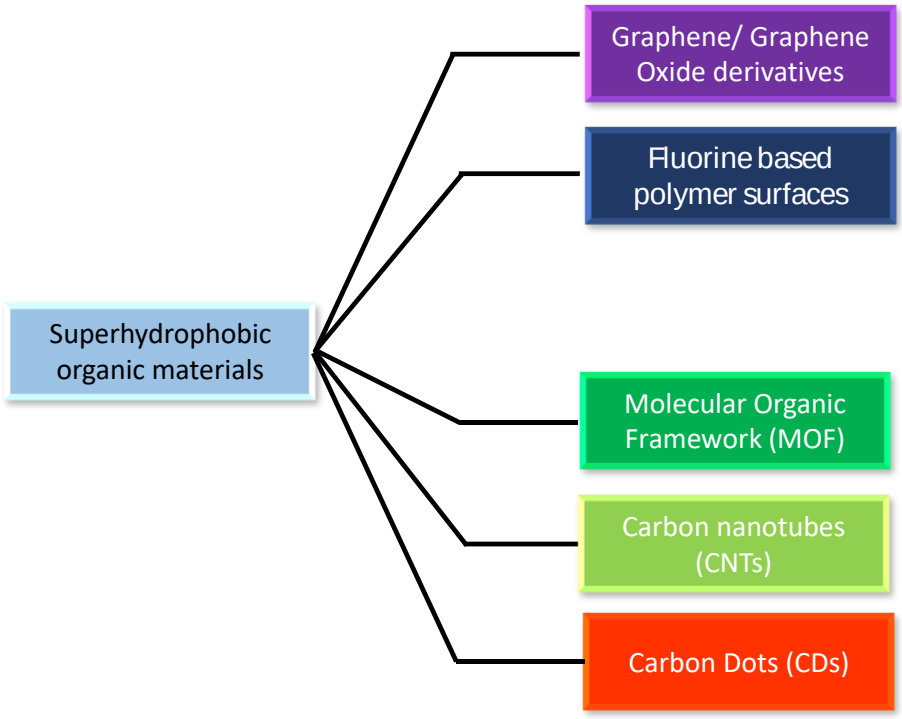


Fig. 1. 1 Superhydrophobic organic materials.

Superhydrophobic surfaces are frequently observed in natural settings, for instance, on the foliage of the lotus plant, the wings of the butterfly, and the body parts of the water strider. The observation of natural phenomena has served as a stimulus for the creation of artificial superhydrophobic surfaces, which exhibit promise for utilization in diverse domains, including but not limited to self-cleaning surfaces, anti-icing surfaces, and microfluidic devices.

The fundamental aspect of producing a superhydrophobic surface lies in the deliberate design of a surface topography that exhibits micro/nano-scale roughness. The formation of this particular texture can be achieved through diverse techniques, including lithography, etching, or nanoparticle coating. Surface chemistry plays a crucial role in the enhancement of water-repellent properties, as the application of a hydrophobic coating can be employed for this purpose.

The hydrophobic characteristics of a superhydrophobic surface can be augmented by incorporating hierarchical structures that integrate micro-

and nano-scale topographies. The presence of a hierarchical structure affords the water droplet with several length scales to engage with, leading to enhanced hydrophobicity.

1.3.1 Characteristics of superhydrophobic surfaces

Wetting hysteresis:

Superhydrophobic surfaces display wetting hysteresis, which refers to the variation between the advancing and receding contact angles of a water droplet. The contact angle of a droplet exhibits variability contingent upon its deposition or removal from the surface. The measurement of wetting hysteresis magnitude is a crucial parameter in the development of superhydrophobic surfaces, as it governs the droplet's ease of sliding off the surface.

Durability:

The durability of a superhydrophobic surface is a crucial factor to be taken into account for practical implementations. The water-repellent properties of superhydrophobic surfaces may deteriorate over time as a result of mechanical abrasion, chemical decomposition, or exposure to environmental conditions. Various coating materials and surface modification techniques are being investigated by researchers to enhance the durability of superhydrophobic surfaces.

Adhesion:

The phenomenon of adhesion is a pertinent issue in the context of superhydrophobic surfaces, which despite their ability to repel water, may still exhibit an ability to adhere to other liquids or particles. The phenomenon of oil droplets adhering to a superhydrophobic surface can be attributed to their relatively low surface tension. Likewise, the deposition of dust or other particulate matter onto the surface may impede its hydrophobic characteristics. In order to tackle these aforementioned obstacles, scholars are currently in the process of

creating surfaces that possess either omni phobic or anti-fouling characteristics.

Self-cleaning:

Superhydrophobic surfaces are commonly recognized as "self-cleaning" due to their ability to effectively eliminate dirt or other impurities from the surface as water droplets effortlessly roll off. The utilization of this technology exhibits potential implications in various industries, including architecture, wherein the implementation of self-cleaning building materials could potentially mitigate the necessity for maintenance.

Fabrication techniques:

The fabrication of superhydrophobic surfaces can be achieved through various techniques such as chemical vapor deposition, plasma treatment, and laser ablation. The selection of a particular methodology is dependent upon the intended surface texture, coating substance, and intended use.

Surface energy:

The determination of the wetting properties of a material is significantly influenced by its surface energy. Surfaces that exhibit superhydrophobicity are characterized by a significantly reduced surface energy, resulting in a high contact angle and a low degree of adhesion between the surface and water droplets. Surface functionalization is a process that can be employed to modify the surface energy of a material. This technique involves the introduction of chemical groups onto the surface of the material, which can alter its properties.

Surface tension:

The cohesive force between water molecules and other materials is attributed to the surface tension of water. Superhydrophobic surfaces diminish the effective surface area of water that interfaces with the surface, thereby decreasing the adhesion force between the water and the surface. Understanding the function of surface tension in wetting phenomena is a crucial aspect in the development of superhydrophobic surfaces.

Applications:

Superhydrophobic surfaces possess an enormous number of potential applications across diverse fields. Superhydrophobic coatings have the potential to decrease the drag on automobiles within the automotive sector by inhibiting the adhesion of water droplets onto their exteriors. Superhydrophobic coatings have potential applications in the medical sector, specifically in mitigating biofouling and bacterial adhesion on medical implants. Superhydrophobic coatings have potential applications in the oil and gas sector for mitigating corrosion and fouling on pipes and equipment. The utilisation of superhydrophobic surfaces is progressively broadening due to the development of novel materials and fabrication methodologies.

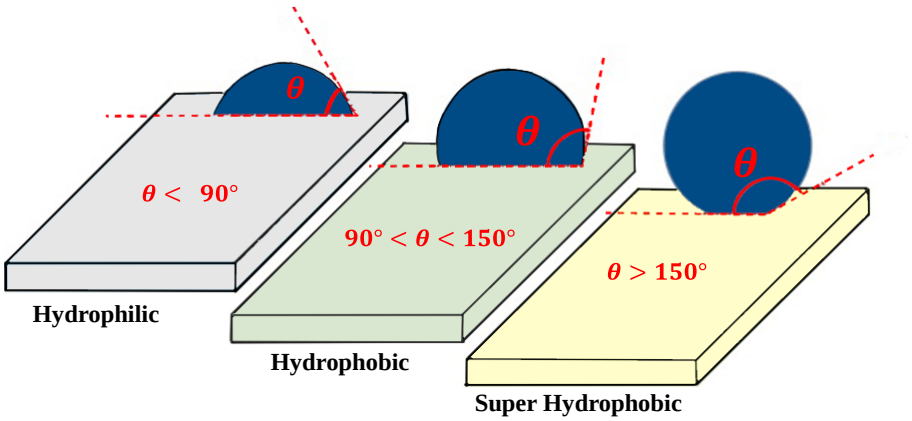


Fig. 1. 2 Nature of surfaces based on contact angle.

Higher water contact angle values suggest that a water droplet tends to keep a spherical form at the surface. Alternatively, a lower water contact angle indicates the movement of a water droplet to unfold at the surface.

1.4 Motivation

Advancements in Materials Science: Hydrophobic fluorescent carbon dots are an exciting and quickly expanding field in the science of materials. Researchers have the potential to contribute to the development of new materials that possess unusual characteristics if they investigate the synthesis, characterisation, and uses of these materials. Because of this, researchers are inspired to dive further into synthesis approaches, attempt to comprehend the fundamental concepts that underlie them, and investigate the prospective applications of these methods.

Combination of Desirable Properties: Carbon dots with a combination of hydrophobicity and fluorescence are known as hydrophobic fluorescent carbon dots. This property offers up a wide range of fascinating possibilities for use in a variety of applications. The impetus for this research comes from the need to understand and make use of the complementary impacts that these qualities have on one another in order to develop functional materials with increased capabilities. This novel

combination has the potential to lead to the development of applications in a wide variety of fields, including sensing, imaging, medication delivery, environmental monitoring, and more.

Fingerprint Analysis and Forensic Science: The combination of hydrophobicity and fluorescence in carbon dots has the potential to have substantial repercussions for the field of forensic science, notably in the area of fingerprint analysis. The need to create more refined methods of fingerprint detection and recognition is the source of the drive for this research. Researchers have shown that they may improve the visualization, sensitivity, and accuracy of fingerprint analysis by making use of the hydrophobicity and fluorescent features of carbon dots. This can be of assistance in criminal investigations and forensic analysis.

Surface Coatings and Functional Surfaces: The creation of hydrophobic surfaces has attracted a lot of interest owing to the fact that these surfaces can clean themselves, are resistant to corrosion, and do not accumulate fouling. The purpose of this research is to investigate the feasibility of using hydrophobic fluorescent carbon dots as building blocks for developing superhydrophobic coatings and functional surfaces. The researchers want to understand the processes that are behind the water-repellent behavior, optimize the features of the coating, and create new applications in industries such as coatings, textiles, and biomedical devices.

Environmental and Biomedical Applications: Hydrophobic fluorescent carbon dots have a number of opportunities for use in the realms of biomedical research and environmental monitoring. The need to find solutions to problems, including the detection of pollutants, the evaluation of water quality, and bioimaging, is the motivation behind this research. Researchers are eager to investigate the sensing, monitoring, and diagnostic applications of these materials in the hopes of developing solutions that are effective, economical, and kind to the environment.

Sustainable and Green Materials: The ever-increasing need for sustainable and environmentally friendly materials is the inspiration behind the research being conducted on hydrophobic fluorescent carbon dots. As an ecologically acceptable alternative to traditional materials, carbon dots may be manufactured from low-cost and numerous carbon sources, such as biomass or waste materials. This enables them to be produced in large quantities. Their hydrophobic and luminous features add value to their sustainability, as does the fact that they may be used in applications that are ecologically aware, as well as devices that save energy and applications that detect pollutants. Researchers contribute to the creation of eco-friendly materials and promote the ideas of green chemistry and sustainable practices by concentrating on hydrophobic fluorescent carbon dots. This helps to promote sustainable practices.

1.5 Research gap

There is still a large knowledge gap in the particular field of hydrophobic fluorescent carbon dots and their implementation in practical settings, despite the rising interest in carbon dots and the numerous applications to which they might be put. The majority of the research that has been done thus far has concentrated on either hydrophobic carbon dots or fluorescent carbon dots separately. However, the investigation of their combined hydrophobic and fluorescent capabilities has only reached a limited level of depth.

There has not been a lot of research put into the synthesis and characterization of hydrophobic fluorescent carbon dots, thus, there is potential for further research into furthering our knowledge of their characteristics and making them more effective. The vast majority of research has concentrated, for the most part, on traditional hydrophobic materials or complicated fluorescent systems, but very little attention has been paid to the potential of hydrophobic fluorescent carbon dots as a separate and adaptable material.

In addition, there is a lack of in-depth study on the many uses of hydrophobic fluorescent carbon dots. Even though fingerprint analysis and superhydrophobic coatings have been discovered as promising applications, not a lot of studies has been done to investigate and find ways to improve these uses of hydrophobic fluorescent carbon dots. More research has to be done in a number of different areas, including the creation of methods for fingerprint analysis that are effective, sensitive, and dependable, as well as the investigation of sophisticated superhydrophobic coatings that can be created utilizing carbon dots.

Furthermore, there hasn't been a lot of research done on how hydrophobic fluorescent carbon dots may be used in the medicinal and environmental fields. Studies are required to study the potential of these carbon dots in a variety of fields, including the detection of pollutants, the evaluation of water quality, bioimaging, and the development of medication delivery systems. In order to fully use their potential for applications in biomedicine and sustainable practices, it is essential to have a solid grasp of their characteristics and how they act in a variety of settings.

In conjunction with the research gap in hydrophobic fluorescent carbon dots, there is also a considerable gap in the investigation of solid-state fluorescent hydrophobic carbon dots. This gap is crucial since there is a correlation between the two fields. The exploration of carbon dots' solid-state characteristics and applications is still very restricted, despite the fact that the majority of the available research literature focuses on forms of carbon dots known as solutions or suspensions.

When compared to its counterparts in the liquid state, fluorescent hydrophobic carbon dots in the solid form provide a number of distinct advantages as well as prospective applications. Techniques like as thin film deposition, surface functionalization, and embedding inside a solid matrix are required for the production of solid-state carbon dots. However, in order to optimize the preparation processes, know the

structural changes, and improve their fluorescence and hydrophobicity in the solid form, these methods will need more research.

Another significant field of study is the characterization of solid-state fluorescent hydrophobic carbon dots. X-ray diffraction, solid-state nuclear magnetic resonance (NMR), and electron microscopy are all examples of techniques that may give insightful information on the crystal structure, surface morphology, and composition of these solid-state materials. It is essential to have an understanding of their properties while they are in the solid state in order to customize their characteristics and optimize their performance for certain applications.

In addition, a significant portion of the potential uses for solid-state fluorescent hydrophobic carbon dots has not yet been investigated. These materials have a significant amount of untapped potential in many different application areas, including optoelectronics, light-emitting devices, sensor technology, and solid-state lighting. Researchers are able to design novel applications that provide increased stability, endurance, and compatibility with a variety of substrates by making use of the fluorescence and hydrophobic features of the materials in their solid form.

For the purpose of developing materials science and broadening the scope of possibilities in a variety of sectors, it is vital to conduct research into the synthesis, characterisation, and applications of solid-state fluorescent hydrophobic carbon dots. By bridging this research gap, we will be able to make a contribution to the creation of solid-state materials with increased fluorescence and hydrophobicity, which will in turn open up new doors for technical developments and practical application.

Chapter 2

Literature Review

2.1 Solvothermal process

In the study conducted by Yan Huo et al., they explored the use of solvothermal methods for the synthesis of crystalline materials at relatively low critical temperatures. Solvothermal synthesis involves using organic solvents heated above their boiling points to increase the pressure inside the reaction vessel. This method allows for precise control over reaction parameters, resulting in high product purity and reduced agglomeration.

One of the key advantages of solvothermal synthesis is its ability to produce carbons with unique physical attributes and excellent configuration specificity. By adjusting factors such as temperature, pressure, solvent type, and other variables, researchers can modify the properties of the resulting carbon materials. This includes controlling particle dispersion, crystallinity, and purity. These adjustments provide flexibility in tailoring the carbon materials for specific applications [1].

Moreover, solvothermal methods offer several benefits for synthetic and doped carbon production. The low critical temperatures required in solvothermal synthesis contribute to the eco-friendliness and cost-effectiveness of the process. It enables the use of organic matter as a precursor, which is widely available and often less expensive compared to other carbon sources. This broadens the scope of materials that can be utilized in carbon synthesis, leading to a more sustainable and economically viable approach [2].

Another significant advantage of solvothermal techniques is their suitability for producing highly crystalline structures. Crystallinity is desirable in many applications, as it can enhance the material's

properties and performance. The controlled reaction conditions in solvothermal synthesis promote the growth of well-defined crystalline structures, resulting in materials with improved structural integrity and specific functionalities [3][4].

2.2 Superhydrophobicity

Peng et al. presented a study The objective of this study was to develop a durable and robust superhydrophobic coating that can be applied to various surfaces through a spray-on method, which was successfully achieved by the researchers. The creation of the coating was achieved through the utilization of a technique that is both cost-effective and easily scalable [5].

This study focuses on the development of a coating through the integration of functionalized silica nanoparticles into a siloxane-modified epoxy nanocomposite. The modification of epoxy using an amino-functionalized polysiloxane was conducted prior to the addition of nanoparticles. The performance of the coating was enhanced through the modification process in comparison to a coating produced using raw epoxy. The experimental results demonstrate that the surface modification technique employed in this study effectively enhances the hydrophobicity of the surface, as evidenced by the increase in contact angle from 153° to 165° and the decrease in sliding angle from 6° to 3°.

The effect of silica concentration in the nanocomposite was studied by the researchers. The optimal balance between superhydrophobicity and durability was achieved at a concentration of around 28% by weight, as determined by the researchers. The hierarchical structure of a coating's surface, which is responsible for its water-repellent properties, can be achieved through the promotion of small growths and spherical microfeatures. The presence of sufficient binding polymer in the coating is crucial for maintaining its structural integrity [6].

The influence of solvent volatility was investigated by the researchers. The study found that utilizing acetone as a solvent in the spray emulsion resulted in an increase in contact angle from 154° to 165° and a decrease in sliding angle from 9° to 3° in comparison to the use of ethanol. The impact of solvent volatility on surface microstructure formation resulted in differences in the wetting performance of the coating [7].

The purpose of this study was to evaluate the performance and durability of coated samples through various tests. This study shows that the coating possesses remarkable adhesion strength and self-cleaning properties when applied on various substrate materials, including textiles. The coating's ability to maintain a contact angle above 150° and a sliding angle below 10° was demonstrated through abrasion tests conducted over 120 cycles. The effectiveness of the material was maintained even when subjected to harsh conditions such as exposure to corrosive environments with a pH range of 1 to 13 and high temperatures of up to 150°C for a duration of 3 hours [8].

Parvate et al. presents a comprehensive survey of recent developments in superhydrophobic coatings. The coatings have been specifically engineered to exhibit hydrophobicity and have garnered considerable attention owing to their distinctive characteristics [9].

Inorganic materials, particularly metal oxides such as ZnO, TiO_2, SiO_2, and clay, have been widely employed as precursors for the development of superhydrophobic coatings. These materials possess both benefits and drawbacks. Zinc oxide (ZnO) and titanium dioxide (TiO_2) have exhibited remarkable efficacy owing to their varied nanoscale architectures, adaptability to diverse manufacturing methods, economical viability, and eco-friendly characteristics [10].

Conversely, organic substances, notably fluoropolymers, have a notable impact on superhydrophobic coatings. Fluoropolymers possess unique benefits in comparison to inorganic substances. These materials are

characterized by their ease of fabrication, scalability for mass production, structural flexibility, and elastic properties [11].

Siloxane-incorporated polymers have been identified as a significant substitute for fluoropolymers. The presence of siloxane groups in these polymers confers exceptional properties such as chemical inertness, thermal stability, elevated electrical resistance, and hydrophobicity.

2.3 Synthesis of the Hydrophobic carbon dots

Yang et al. synthesized hydrophobic carbon dots (H-CDs) that display reversible two-switch-mode luminescence, namely blue dissolved fluorescence and red aggregation-induced emission (AIE). The luminescent properties were attained by the authors via a one-pot solvothermal treatment that is both eco-friendly and cost-effective [12].

The researchers employed various techniques, including transmission electron microscopy (TEM), X-ray diffraction (XRD), and Raman spectroscopy, to verify the characteristics of the H-CD carbon nanoparticles. The H-CDs were found to possess a hydrophobic disulfide bond featuring a symmetrical heterocyclic surface, as evidenced by X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FT-IR), and nuclear magnetic resonance (NMR) analyses. The hydrophobic nature of the H-CDs is explained by a large number of pyridinic and epoxy molecules on their symmetrical heterocyclic surfaces.

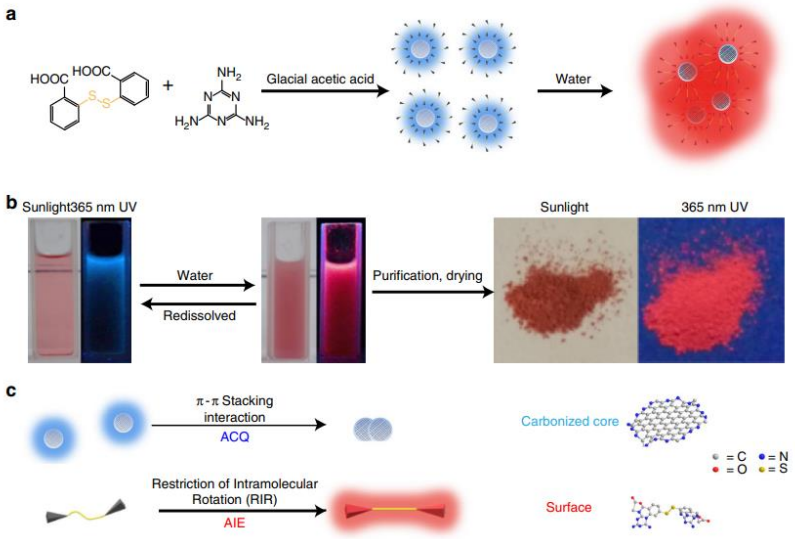


Fig 2. 1 a) The generation of H-CD monomers and their conglomerates is being investigated, with emphasis on the disulfide bond present in the dithiosalicylic acid molecule, which is visually marked in yellow. b) Photographs depicting the luminescence principle of the H-CDs' dual-switch mode. c) The principle of fluorescence and the suggested configuration of the core and surface of H-CD are depicted, with the glowing edges corresponding to the hue of their fluorescence [13].

The H-CDs, upon being dispersed as monomers in organic solvents such as acetone and ethanol, exhibited conventional ultraviolet absorption and blue emission. The hydrophobicity of the H-CDs was observed to be high, which can be attributed to the existence of epoxy and pyridyl functional groups on their surfaces. As a result, upon the introduction of water, the H-CDs underwent precipitation. Upon precipitation, the hydrogenated carbon dots (H-CDs) underwent J-aggregation, leading to aggregation-caused quenching (ACQ) of their carbon cores. This phenomenon was attributed to the π-π stacking interactions within the H-CDs' extensive conjugated system. Consequently, the blue emission was forfeited. The surface energy transition that was dominant underwent a change, leading to the emergence of red aggregation-induced emission (AIE) upon restricting the intramolecular rotation of symmetrical heterocycles around the disulfide bonds [14].

The reversible two-switch-mode luminescence mechanism was substantiated by the previously mentioned characterization techniques, along with experiments carried out on H-CD monomers and aggregates in a dimethylformamide (DMF) solvent. The experimental findings indicate that the pure H-CD monomers dissolved in AC or ethanol solutions displayed exclusive blue emission, whereas the pure H-CD powder manifested solely red aggregation-induced emission. The coexistence of monomers and aggregates in a DMF solution resulted in the observation of both blue-dissolved fluorescence and red aggregation-induced emission (AIE) [12].

Two experimental controls were performed to provide additional confirmation of this theory, verifying the reversible transition between blue emission and red aggregation-induced emission (AIE) in the hydrogenated carbon dots (H-CDs). The researchers utilized the dual-switch-mode luminescence of H-CDs to produce an advanced fluorescence ink for the purpose of anticounterfeiting and dual encryption [15].

Yin et al. discusses the synthesis and characteristics of hydrophobic carbon dots that are derived from organic molecules featuring a singular functional group, such as aliphatic amine and aliphatic aldehyde. The authors emphasize that this discovery broadens the spectrum of precursor materials employed in carbon dot investigations, as earlier techniques required substances with diverse and varied functional groups [16].

The carbon dots were synthesized via pyrolysis, a technique that involves exposing organic molecules to moderate temperatures without the requirement of supplementary reagents. This method provides a convenient approach for the synthesis of hydrophobic carbon dots [17].

Subsequent to the pyrolytic process, the carbon dots that have been synthesized can undergo additional refinement through the utilization of

either gel permeation chromatography (GPC) or silica gel column chromatography. The previously described purification process serves the purpose of eliminating impurities and achieving the isolation of carbon dots, thereby facilitating their subsequent characterization and application.

The carbon dots that were produced demonstrate blue emission, and their photoluminescence (PL) properties are contingent upon the excitation wavelength. The solubility of carbon dots in diverse organic solvents renders them appropriate for utilization as fluorescent inks in various applications [18], [19].

The carbon dots exhibit a noteworthy characteristic in the form of alkyl chains that facilitate the segregation of photoluminescent centers. The process of segregating facilitates the prevention of fluorescence quenching, thereby enabling the carbon dots to retain their fluorescence even in the absence of a solvent.

Zhao et al. define the methodology employed for the synthesis of hydrophobic carbon dots (CDs) that exhibit yellow luminescence. The previously mentioned outcome was attained through the introduction of a dehydrated agent, DCC, into a blend of two carbon substrates, specifically L-cysteine hydrochloride (L-cys·HCl) and citric acid (CA). The hydrophobic CDs that were produced demonstrated a significant quantum yield of 30%, thereby indicating their efficacy in terms of light emission [20].

The impact of various synthesis parameters on the optical properties of hydrophobic CDs was also examined by the researchers. The researchers employed diverse characterization methodologies to comprehend the optical characteristics and surface functionalities of the CDs, with the objective of investigating the underlying mechanism responsible for their luminescent behavior [21].

Furthermore, the investigators proceeded to encapsulate the hydrophobic CDs that had been prepared within liposomes. The aforementioned procedure resulted in the conversion of the hydrophobic CDs to hydrophilic CDs, thereby rendering them water-soluble. The aforementioned alteration expands the potential applications of hydrophilic CDs.

The researchers investigated the prospective uses of hydrophobic and hydrophilic cyclodextrins in various domains. The researchers exhibited that hydrophobic cyclodextrins can serve as solid luminescent materials with the ability to be shaped into specific forms. Furthermore, the researchers examined the viability of utilizing hydrophobic CDs as a source of illumination for LED lighting fixtures [22].

A salient feature of this study is that the production of hydrophobic substances was carried out through a solid-phase synthesis approach. The utilisation of this technique obviates the necessity for organic reagents and notably diminishes the reaction duration requisite for the acquisition of CDs that emit in the long-wavelength range. The aforementioned approach confers benefits by streamlining the process of synthesis and facilitating the efficient generation of hydrophobic CDs with elevated quantum yields [23].

The researchers, led by Susanta Kumar Bhunia, conducted a study on the production of functional nanoparticles using red fluorescent carbon nanoparticles. The team also evaluated the potential of these nanoparticles as a fluorescent label for cells. The carbon nanoparticle that is functional exhibits a hydrodynamic size of less than 25 nm, possesses high colloidal stability when subjected to physiological conditions, and can be stimulated using blue and green light to capture its red emission. The transformation of polyethylene glycol and primary amine terminated carbon nanoparticles into desired nano bioconjugates can be achieved through the utilization of commercially available reagents and protocols [24].

Chapter 3

Materials and Instruments

3.1 Chemicals and Materials

Methanol (CH_3OH) was purchased from Avantor performance material India Ltd., Thane, Maharashtra (India). Acetic Acid (CH_3COOH) was obtained from Sisco Research Laboratories Pvt. Ltd. (Mumbai, India). HBT (2-Hydrazinobenzothiazole >99.0%) and DBA (2,2´-Dithiodibenzoic Acid >96.0%) were purchased from TCI Chemicals (India), Chennai (India). ATA (4-Amino-4H-1,2,4-triazole - 99%) was purchased from Avra synthesis Pvt. Ltd. All the reagents were used as received without further purification.

3.2 Instrument and Characterization

FT-IR data were obtained using spectrum two PerkinElmer ATR FT-IR spectrometer, UV-vis study was done using UV2600 Schimazdu spectrophotometer. A JOEL7610F Plus was used to take FE-SEM pictures. The ^1H NMR spectra were obtained using a Fourier Transform Nuclear Magnetic Resonance spectrometer (Model AVNACE NEO500 Ascend Bruker BioSpin International AG, Switzerland).

Chapter 4

Synthesis

4.1 Material Synthesis

4.1.1 BCD

Carbon dots were synthesized via a one-pot solvothermal method utilizing melamine and L-cystine as precursor materials and acetic acid as the solvent, followed by a straightforward purification step. It is noteworthy that acetic acid plays a crucial role in the formation of carbon dots. Furthermore, this solvent exhibits eco-friendliness and cost-effectiveness while also serving as a catalyst for carbon dots. A solution containing 100.8 mg of melamine and 384.5 mg of L-cystine was subjected to ultrasonic treatment in 20 mL of acetic acid. The resulting solution was then placed in a Teflon reactor with a capacity of 50 mL and heated at 180 °C for ten hours in an oven. The solution that had been prepared underwent centrifugation at a rate of 10,000 revolutions per minute for a duration of 20 minutes, followed by filtration. Upon exposure to ultraviolet radiation, the solution exhibits a pale greenish-white hue.

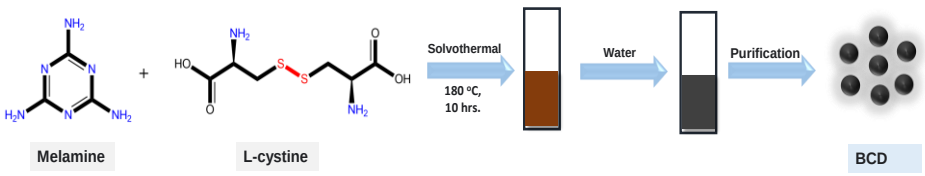


Fig. 4. 1 Carbon dot solution under UV light.

Following the solvothermal process, the resultant solution was mixed with 500 ml of boiled water to generate a solid precipitate and eliminate any remaining unreacted materials and solvents. Subsequently, the powder that was acquired was dispersed onto distilled water, and it was

observed that the powder exhibited hydrophobic properties, as evidenced by the formation of a meniscus on the surface of the water.

4.1.2 CR

A suspension of 132.05 mg HBT (2-Hydrazinobenzothiazole0 and 272 mg DBA (2,2´-Dithiodibenzoic Acid) was prepared using 20 mL acetic acid as solvent. The prepared solution was sonicated in an ultrasonicator to make it miscible. Further, it was transferred into a 50 ml Teflon autoclave and kept in an oven at 150°C for 10 hrs. The impact of reaction parameters, such as temperature, time, the quantity of precursor, etc., was studied.

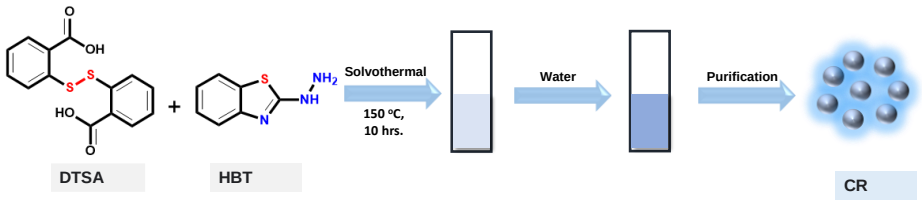


Fig. 4. 2 Synthesis of CR powder.

4.1.3 YR

A suspension of 67.2 mg ATA (4-Amino-4H-1,2,4-triazole) and 272 mg DBA (2,2´-Dithiodibenzoic Acid) was prepared using 20 mL acetic acid as solvent. The prepared solution was sonicated in an ultrasonicator to make it miscible. Further, it was transferred into a 50 ml Teflon autoclave and kept in an oven at 150°C for 10 hrs. The impact of reaction parameters, such as temperature, time, the quantity of precursor, etc., was studied.

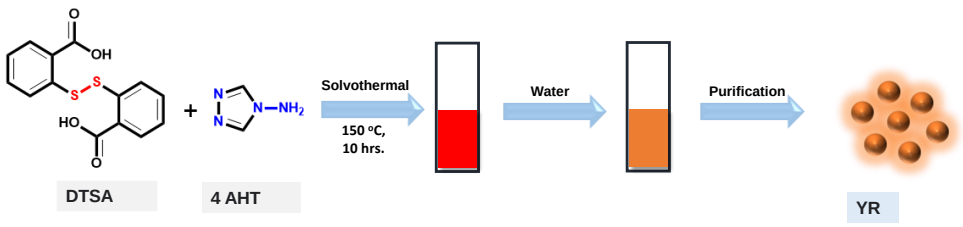


Fig. 4. 3 Synthesis of YR powder.

Chapter 5

Results and Discussion

The objective of using a nitrogen-based precursor and a carbon-based precursor with S-S bonds and a benzene ring in hydrophobic carbon dot synthesis was that Nitrogen-based precursors, such as amines or amides, can provide nitrogen functionalities in the resulting carbon dots. These nitrogen functionalities can contribute to the overall properties of the carbon dots, such as enhancing their photoluminescence or improving their stability.

Carbon-based precursors with S-S bonds introduce sulfur-containing groups into the carbon dots. These groups can contribute to the hydrophobicity and chemical reactivity of the carbon dots. Additionally, they can influence the electronic properties and surface chemistry of the carbon dots, leading to unique applications in catalysis, sensing, or energy storage.

The presence of a benzene ring in the carbon-based precursor can impact the structure and properties of the resulting carbon dots. Benzene rings can participate in aromatic interactions and pi-pi stacking, potentially leading to the formation of more ordered or crystalline carbon dots. This can affect the optical, electronic, and mechanical properties of the carbon dots and broaden their potential applications.

The yield of BCD powder was very less, it showed a whitish fluorescence in liquid state while the fluorescence was completely quenched in liquid state. Moreover, the powder exhibited inferior hydrophobic properties.

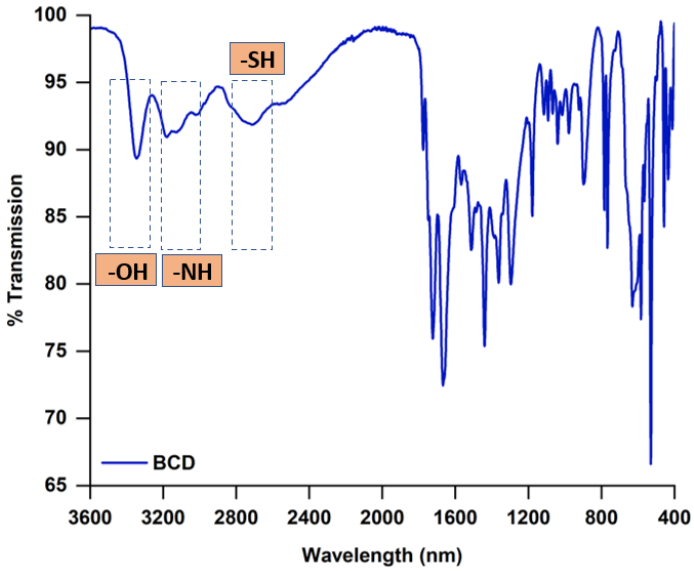
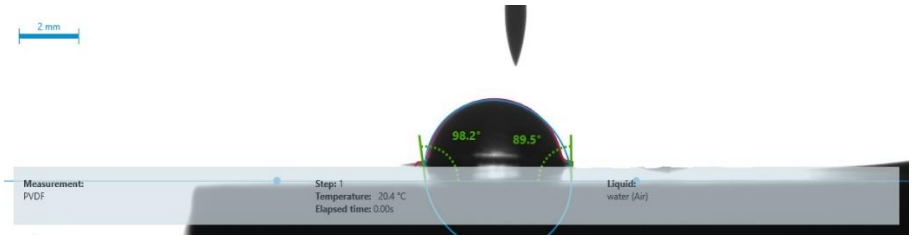


Fig. 5. 1 FTIR spectra of BCD powder.

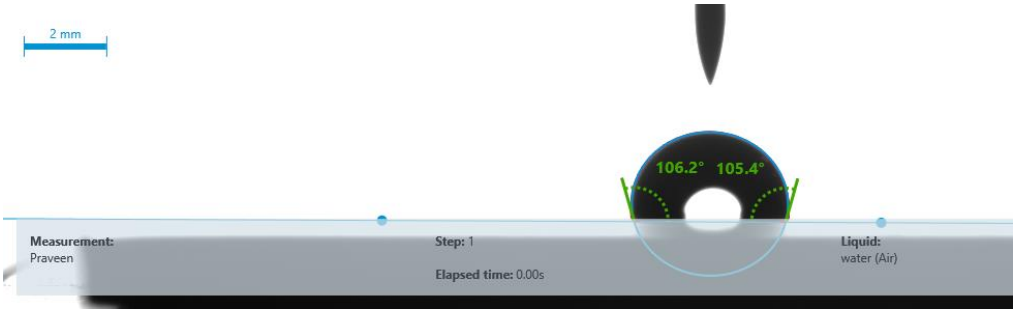
To get the presence of organic and inorganic compounds in the sample. FTIR was performed, which gave the following result. Here we can clearly see the presence of OH functional group which decreases hydrophobicity of the obtained BCD powder.

Further contact angle measurements were done by preparing a coating of BCD powder and PVDF using NMP as a solvent but could not achieve a contact angle greater than 150°.

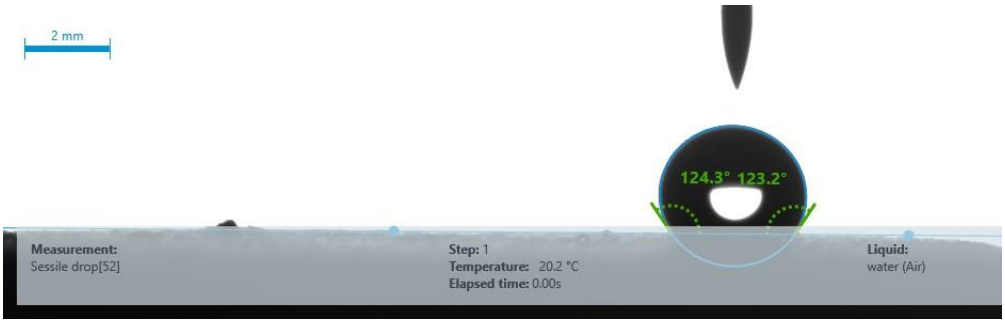
a) Bare sample



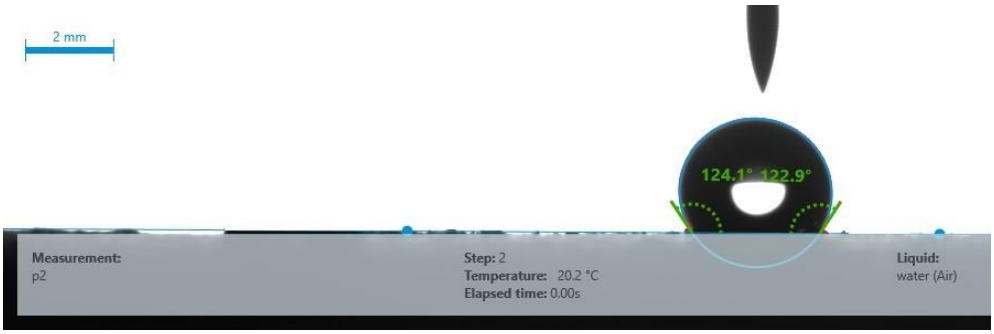
b) PVDF coating



c) PVDF + BCD



d) PVDF + BCD (1:1.5)



e) PVDF + BCD (1:2)

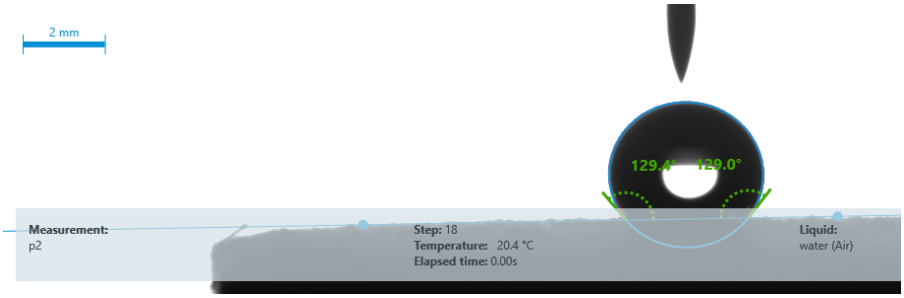


Fig. 5. 2 Contact angle measurements of different coating samples of BCD powder.

Hence, we modified the synthesis strategy by changing the carbon and nitrogen sources and obtained superhydrophobic carbon dots which also exhibited multicolour SSF when precursors were tuned. When an extra aromatic ring was incorporated in the N-source precursor, the solid-state fluorescence of the powder could be drastically tuned from blue to orange and the powders can be utilised to fabricate fluorescent superhydrophobic coatings. Furthermore, the powders were employed to detect latent fingerprints.

5.1 FTIR spectroscopic analysis

FTIR of novel superhydrophobic powders (CR and YR) in different molar ratios

FTIR spectra were used to determine the reason behind the hydrophobicity of the newly obtained powder samples.

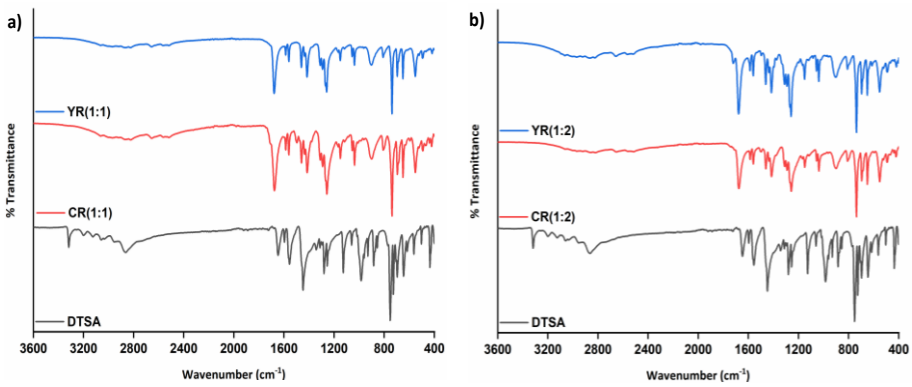


Fig. 5. 3 FTIR data of CR and YR powders under different molar precursor concentrations.

Comparing the spectra of the obtained powder samples with the precursor DTSA, we see that the O-H peak in the precursor at 3300 cm^{-1} was missing in the as-prepared powder samples, which supports the reason behind the hydrophobicity of the powders. Also, we observe the formation of new S-H bonds at 2507 cm^{-1} and S-S bonds at 491 cm^{-1}. Add 1:1 and 1:2 CDs - to examine the effect of changing molar ratios of C-source and N-sources on the presence of the surface groups in the carbon dots, we attempted to optimize the feed ratios- However, as evident from the spectra, peaks positions for -NH, -S-H, >C=O functionalities remained mostly unaltered which implied almost identical surface environment for the CR1, CR2, YR1, YR2 carbon dots. The result was also confirmed by the visual observation under UV light where CR1, CR2 and YR1, YR2 exhibited almost similar solid-state fluorescence.

5.2 UV-Visible Spectroscopic analysis

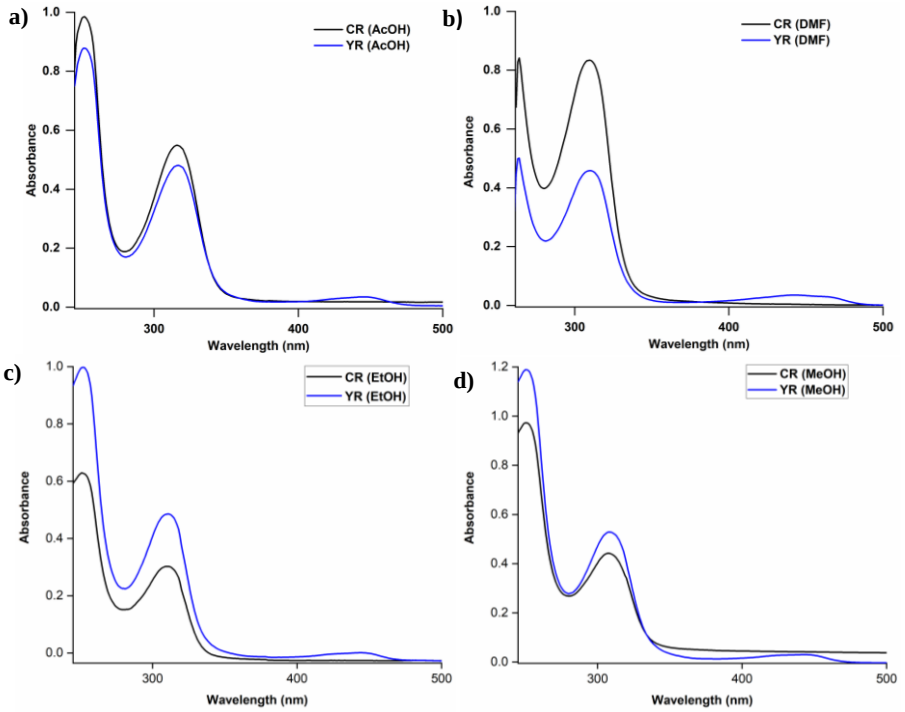


Fig. 5. 4 UV-visible spectroscopy of the as-prepared powder samples in different solvents.

Further, we performed solvent-dependent UV-Vis spectroscopic analysis to assess the optical properties of the synthesized CDs in methanol, ethanol, DMF, and AcOH. These solvents were chosen as the optimum solubility of the CDs was obtained in them. In all solvents, CR-CD showed absorption maxima in the range of 250-275 nm, which attributes to the π-π* transition of C=C/C=N in the sp^2 core of the carbon dot. Beyond this region, peaks in the range of 308-315 nm correspond to the n-π* transition originating from C=N/C=O, C–O and C−S structures. YR-CD exhibits a similar kind of absorption behavior. Interestingly, it exhibits a tail-off region beyond 400 nm, in the range of 410-430 nm, which was absent in CR-CD. This explains the more intense visible color of YR-CD compared to CR-CD.

5.3 Photoluminescence Study

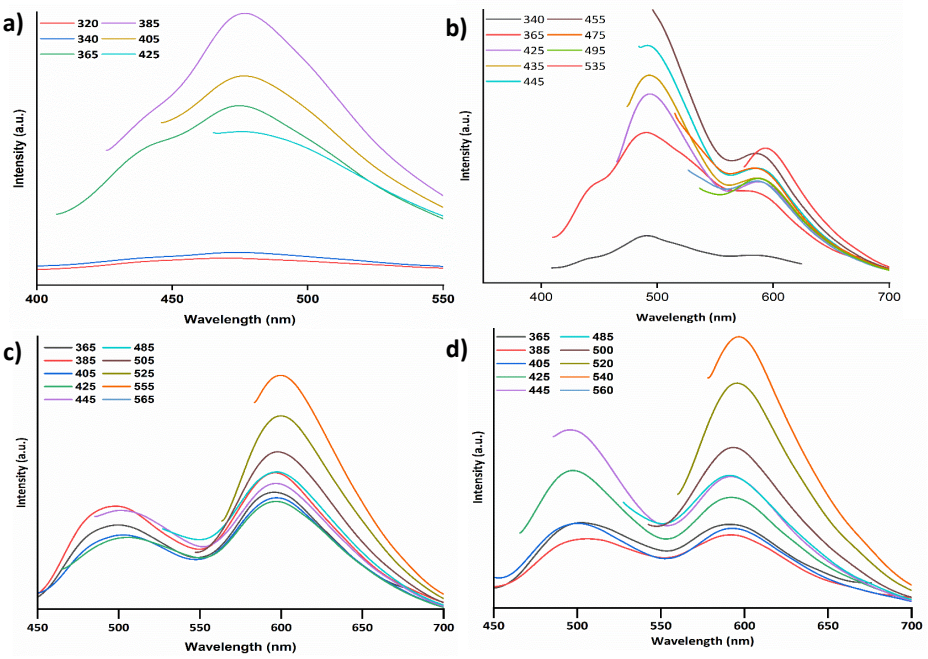


Fig. 5. 5 Photoluminescence Spectra of carbon dots a) CR (1:1), b) CR (1:2), c) YR (1:1), d) YR (1:2).

The fluorescence image demonstrates that the CR and YR carbon dots exhibit bluish white and orange fluorescence in solid-state almost irrespective of their precursor feed ratios, consistent with the PL spectrum depicted above. YR (1:1) and YR (1:2) exhibited two prominent broad peaks located at 500 nm and 593 nm. This observation was affirmed by optically exposing the YR CDs under short and long UV. In case of CR-CDs, both the 1:1 and 1:2 feed ratios exhibited blue fluorescence, with an emission band located at 478 nm. Moreover, it is noteworthy to observe that spectra corresponding to CR-CD and YR-CDs remained almost unaltered in their peak positions regardless of the excitation wavelength within the range of 450-700 nm and 325-450 nm. This suggests that CR and YR demonstrate PL behavior in the solid state that is independent of the excitation wavelength. Although there was almost no difference in the FTIR and fluorescence spectra the superhydrophobic properties were varied to an appreciable level when

the precursor feed ratios were altered which were demonstrated in the later sections.

5.4 Morphological analysis of carbon dots and coating

5.4.1 CR and YR

To obtain the microstructural information of the hydrophobic coating, we performed the FESEM analysis over the freshly prepared samples. FESEM analysis reveals the stacked stick and well-sticked fiber-type microstructure, which might be the reason for the hydrophobicity of the overall sample.

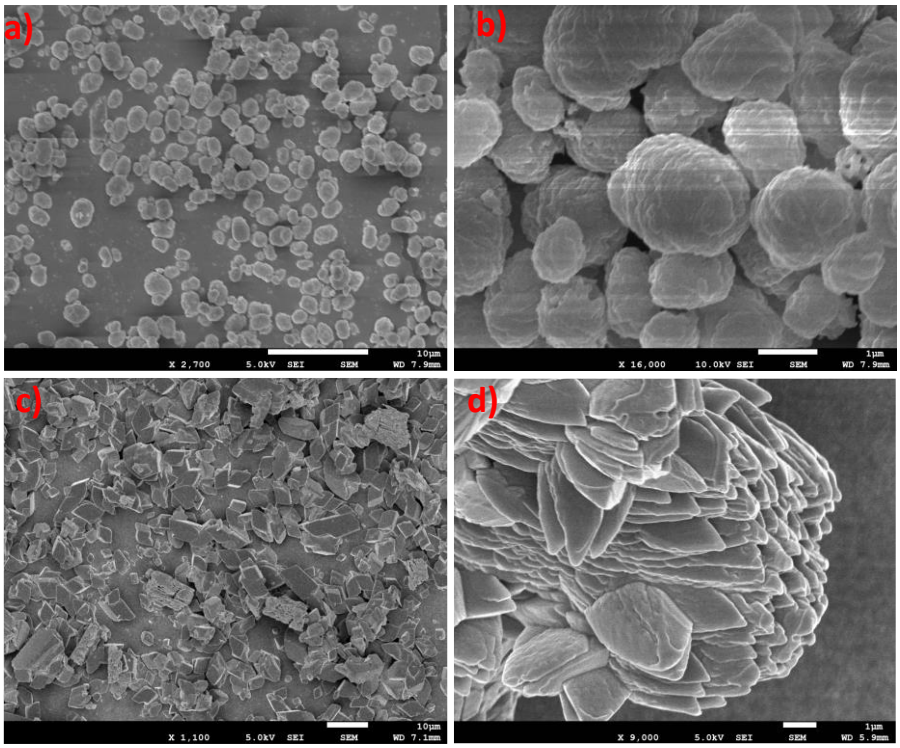


Fig. 5. 6 FESEM image (a-b) CR(1:1), (c-d) CR(1:2) powder samples.

As can be seen from fig, the collected powder of CR (1:1) and CR(1:2) showed prominent differences in morphology, the CR(1:1) showed ball-shaped morphology, and the CR(1:2) powder exhibited flake-like stacked microstructure.

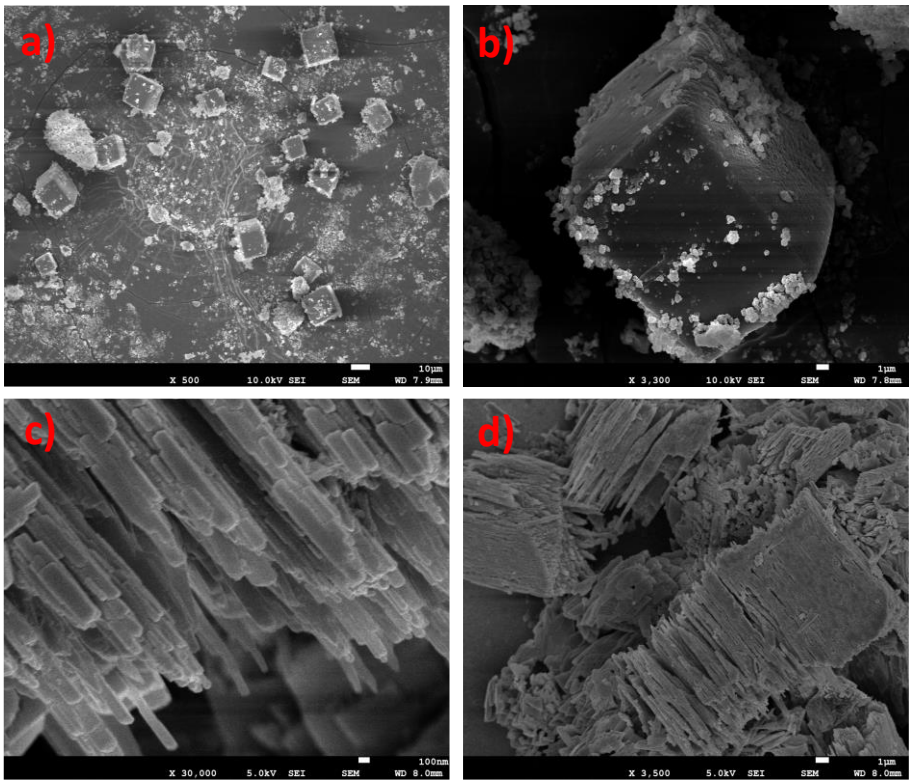


Fig. 5. 7 FESEM image (a-b) YR (1:1), (c-d) YR (1:2) powder samples.

On the other hand, YR (1:1) and YR (1:2) also exhibited distinct differences in morphological characteristics. The YR (1:1) showed cubical structures, and YR (1:2) showed a needle-shaped stacking structure.

Both of these above findings support the prominent alternation in the carbon dots when precursor ratio was changed from 1:1 to 1:2.

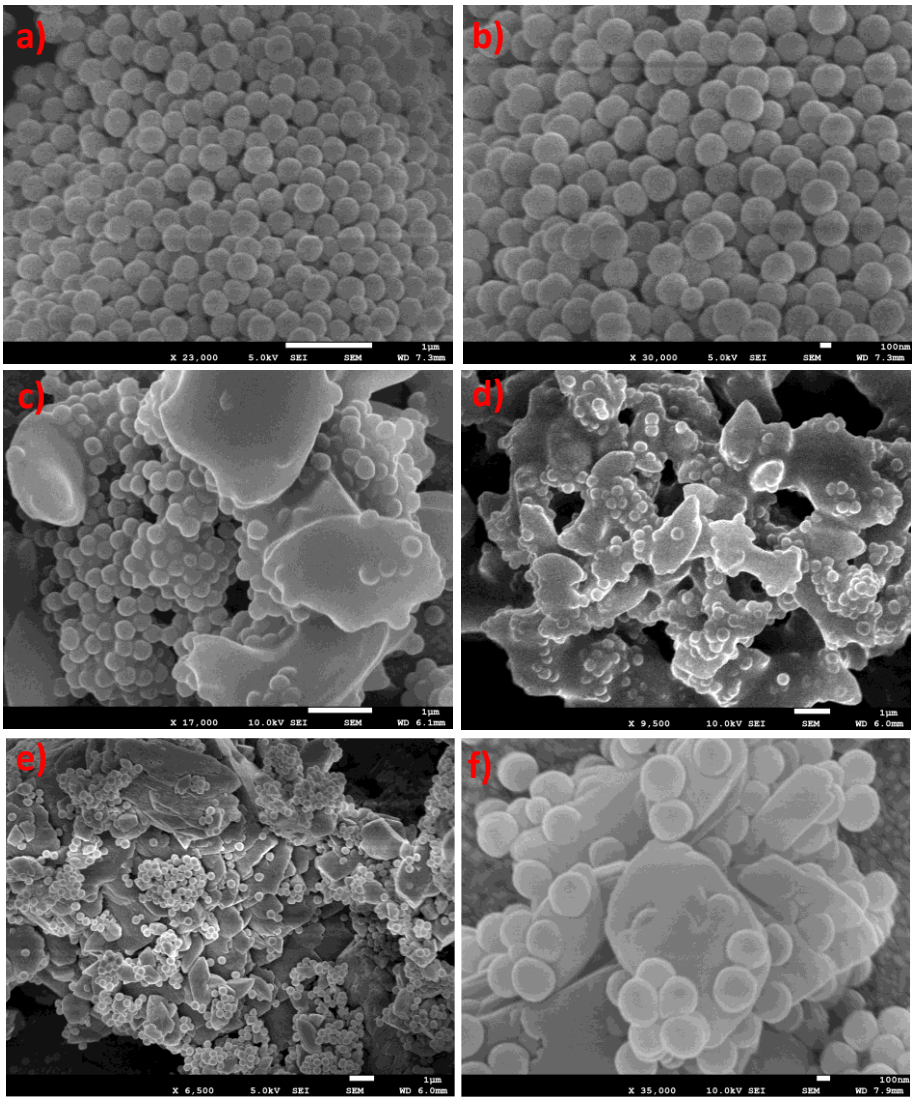


Fig. 5. 8 FESEM image a-b) PVDF coatings, (c-d) PVDF with CR (1:1), (e-f) PVDF with CR (1:2) powder samples.

The morphological characteristics of superhydrophobic coating generated from the corresponding four powders, namely CR (1:1), CR (1:2), YR (1:1), and YR (1:2) we went for FESEM of the powders along with PVDF in methanol.

The PVDF coating showed uniformly distributed large spherulites. In contrast, when the coating was prepared using PVDF and CR (1:1) and YR (1:1) powder, it was found that the uniformly distributed PVDF spherulites were stuck into the CR (1:1) and YR (1:1) matrix, probably

that is why the coating of PVDF alone was easily peeled off. In contrast, the superhydrophobicity and coating stability was largely improved when PVDF and the obtained powders were coated together.

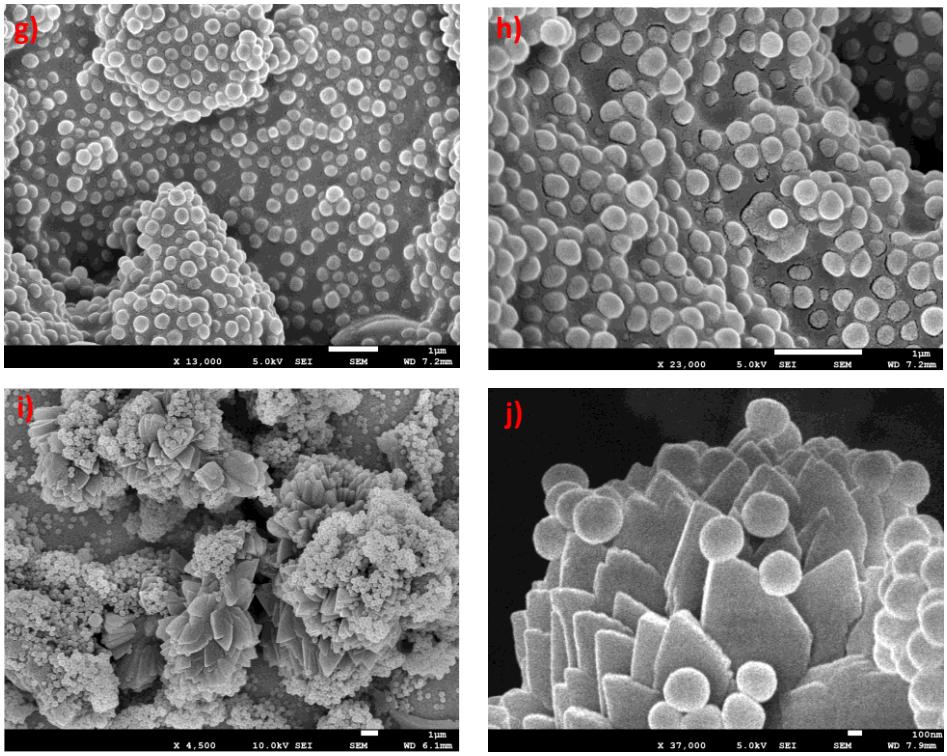


Fig. 5. 9 FESEM image (g-h) PVDF with YR (1:1), (i-j) PVDF with YR (1:2) powder samples.

Interestingly we found fish scale-like morphological growth in CR (1:2) and PVDF coating, where PVDF seems to be deposited along the carbon dots nanostructures, while in YR (1:2) we found petals like morphology where PVDF is being embedded in the spaces between the petals and also on the surface of petals.

5.5 Solubility test for coating optimization

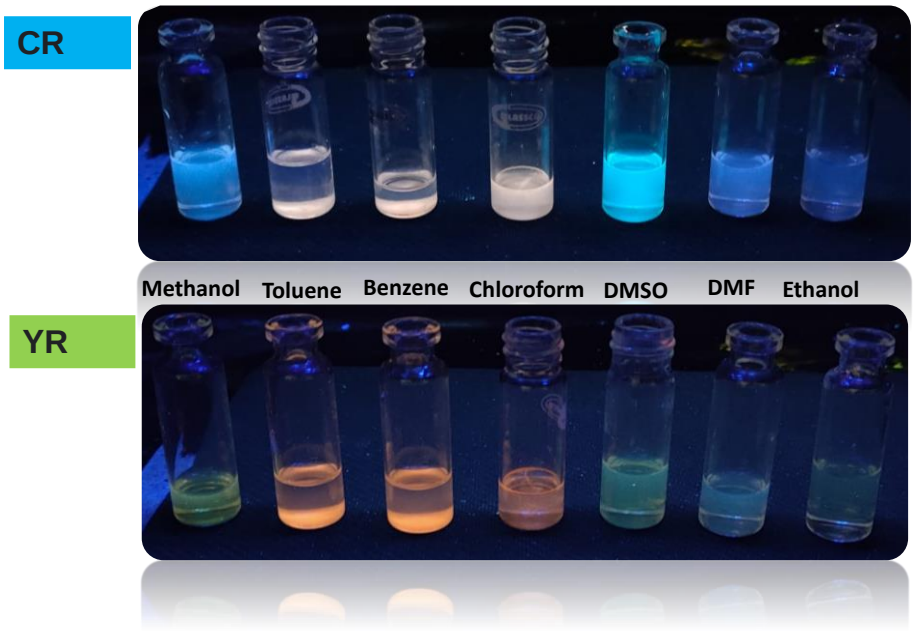


Fig. 5. 10 Test of solubility of CR and YR in different solvents for coating optimization.

- The best solubility of CR and YR was achieved in (i) DMF, (ii) DMSO, (iii) ethanol, and (iv) methanol.
- Benzene, toluene, and chloroform only produced partial dispersion and were ruled out as a solvent to form a stable coating.
- Solvatochromism was also observed for various solvents, and the solution-state fluorescence was quite different from the solid-state fluorescence of the synthesized carbon dots.

5.6 Contact angle

5.6.1 Coatings with novel superhydrophobic materials (CR , YR)

Coatings were prepared using novel superhydrophobic materials (CR, YR) using PVDF as binder and methanol as solvent. Contact angle measurements were performed on the as-prepared coatings; starting with YR (1:1) (a), the contact angle came out to be 160.1°, showing that the coating is superhydrophobic as the contact angle is greater than 150°.

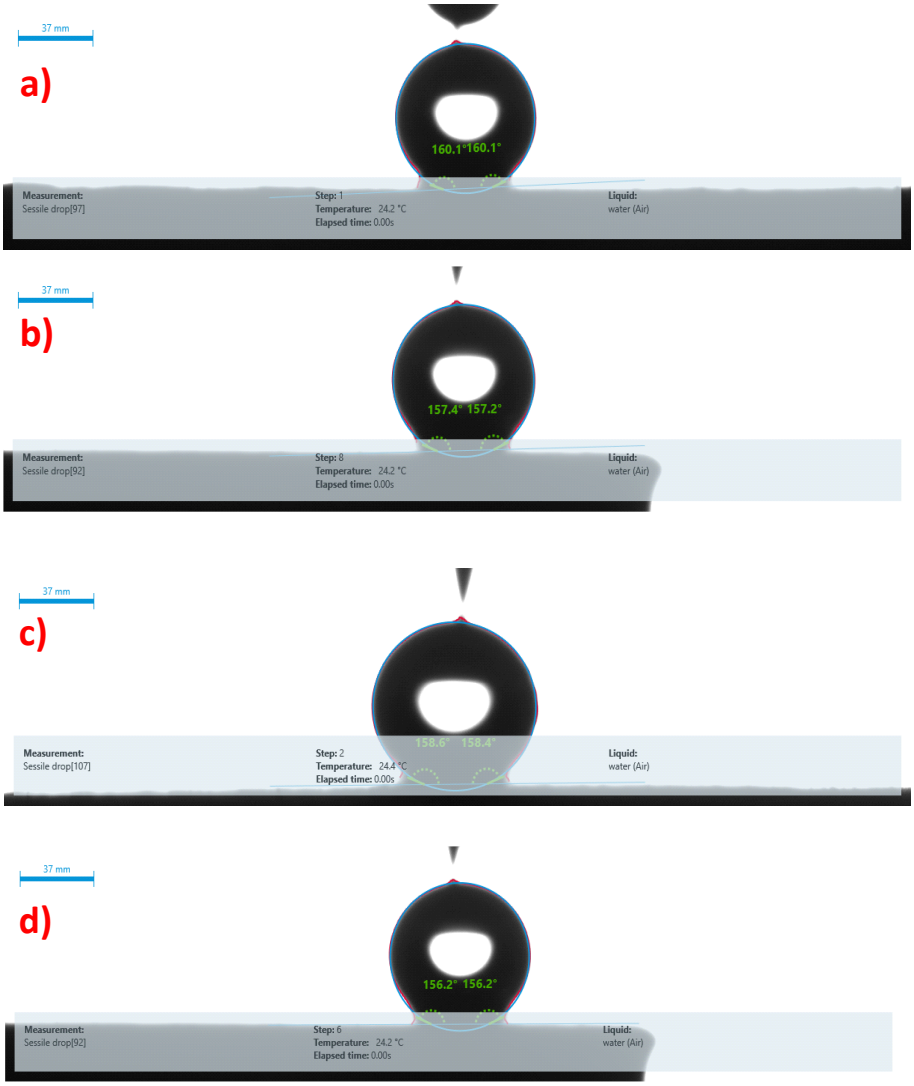


Fig. 5. 11 Contact angle measurement of CR and YR coatings a) YR (1:1), b) CR (1:1), c) CR (1:2), d) YR (1:2).

Similarly, the contact angles of CR (1:1), YR (1:2) and CR (1:2) were 157.4°, 158.6° and 156.2°. The contact angles of all the prepared coatings exceeding 150 degrees indicate that the coatings possess superhydrophobic properties. The fact that the contact angles of the coatings exceeded 150° suggests that the surfaces are highly water-repellent. This indicates that the coatings have achieved the desired superhydrophobic characteristics, which are highly desirable in various applications such as self-cleaning surfaces, anti-fouling coatings, and water-resistant materials.

Chapter 6

Applications

6.1 Solid State fluorescent Carbon dots for Forensic Application: Latent Fingerprint detection using Carbon dot powder

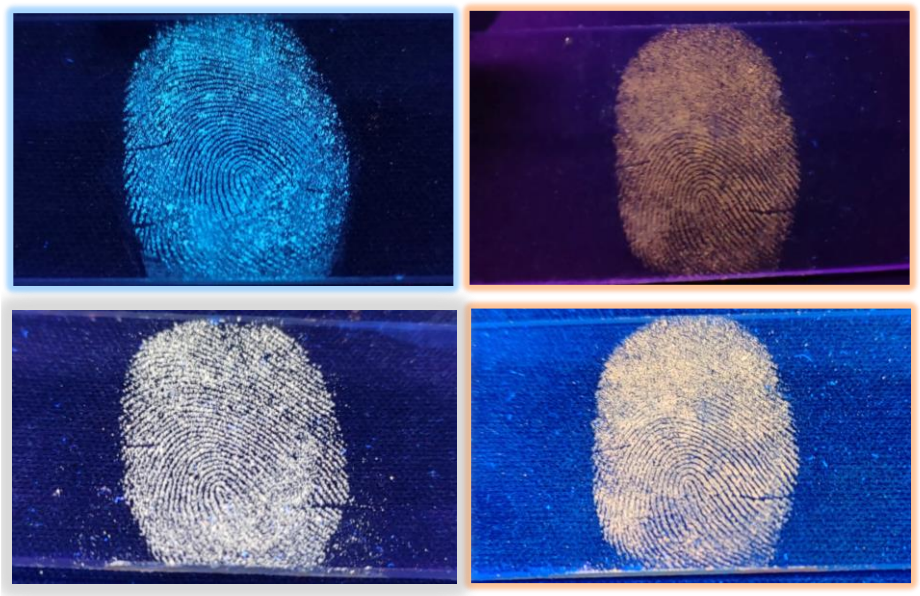


Fig. 6. 1 Fingerprint images under UV light with CR and YR carbon dots.

Fluorescent carbon dots, CR and YR, exhibit promising potential for fingerprint analysis and identification. These tiny nanoparticles possess unique optical properties, allowing them to emit fluorescence when excited by certain wavelengths of light. This fluorescence can be harnessed for fingerprint analysis by incorporating these dots into fingerprint powder formulations.

When CR and YR dots are mixed with fingerprint powder, they adhere to the oily residues present in fingerprints, forming a luminescent coating on the ridges. This coating significantly enhances the contrast between the fingerprint ridges and the surface, making it easier to visualize and capture high-quality fingerprint images. Additionally, the

fluorescent nature of these carbon dots enables the detection of latent fingerprints under different lighting conditions, even in challenging environments.

6.2 Superhydrophobic coating

6.2.1 Sample polishing

Mild steel samples of size 2cm x 2cm has been taken as sample material which will be tested against corrosion. The samples were polished using 80- 800 mesh sand papers.

Fig. 6. 2 Polished sample of Mild Steel.

6.2.2 Prepared superhydrophobic coatings

The process of preparing superhydrophobic coatings on the surface of mild steel using carbon dot powders and polyvinylidene fluoride (PVDF) with methanol as a solvent involves the following steps:

1. Mixing Carbon Dot Powder: Carbon dot powders are combined with PVDF and methanol in a container. The carbon dots serve as the functional material responsible for the superhydrophobic properties of the coating.

2. Ultrasonication: Once the carbon dot powder, PVDF, and methanol are mixed together, the container is placed in an ultrasonicator. Ultrasonication involves subjecting the mixture to high-frequency sound

waves, which create cavitation bubbles. These bubbles collapse violently, generating localized high temperatures and pressures. This process helps in dispersing and homogenizing the components, ensuring proper blending of the carbon dots and PVDF with the solvent.

3. Drop Casting Method: After ultrasonication, the mixture is ready for the application of the superhydrophobic coating. The drop casting method is used for this purpose. In this method, a droplet of the prepared mixture is carefully placed onto the surface of the mild steel substrate.

4. Evaporation: Following the application of the droplet, the methanol solvent begins to evaporate. As the solvent evaporates, the carbon dot powders and PVDF form a thin film on the surface of the mild steel substrate.

5. Formation of Superhydrophobic Coating: As the solvent evaporates completely, the carbon dots and PVDF arrange themselves in a specific manner on the surface, creating a micro/nanostructure. This structure, combined with the low surface energy of the carbon dots and PVDF, imparts superhydrophobic properties to the coating. The resulting coating is highly repellent to water, exhibiting self-cleaning and anti-corrosion characteristics.

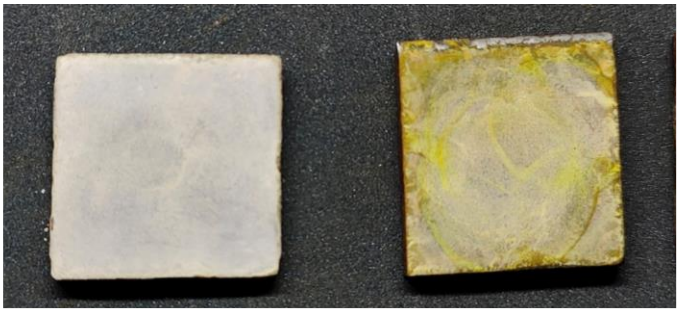


Fig. 6. 3 Superhydrophobic coatings prepared using CR and YR carbon dots.

Chapter 7

Conclusion and Future Scope

7.1 Conclusion

In this report, fluorescent hydrophobic carbon dots were successfully synthesized using the solvothermal method. The carbon dots exhibited both fluorescence and hydrophobicity, as supported by various characterization techniques including FTIR, FESEM, UV-visible spectroscopy, photoluminescence, and contact angle measurements.

The FTIR analysis confirmed the presence of functional groups characteristic of carbon dots, while FESEM images revealed the formation of well-dispersed nanoparticles with a uniform size distribution. UV-visible spectroscopy and photoluminescence measurements demonstrated the absorption and emission properties of the carbon dots, indicating their fluorescence.

Furthermore, the contact angle measurements indicated the hydrophobic nature of the carbon dots, showcasing their potential for superhydrophobic coating applications. The combination of fluorescence and hydrophobicity made these carbon dots suitable for two significant applications: fingerprint detection and superhydrophobic coating.

By utilizing the fluorescence property, the prepared carbon dots were successfully employed in fingerprint detection. The fluorescence of the carbon dots enhanced the visualization and identification of latent fingerprints, facilitating accurate matching and identification processes.

Moreover, due to their hydrophobic nature, the carbon dots were utilized in the development of superhydrophobic coatings. The coatings exhibited excellent water repellency, self-cleaning properties, and

resistance to corrosion. This research opens new possibilities for fingerprint analysis and the creation of advanced hydrophobic surfaces.

7.2 Future Scope

1. Optimization of synthesis parameters: Further investigation into the solvothermal process can focus on optimizing various synthesis parameters such as temperature, reaction time, precursor concentration, and solvent composition. Fine-tuning these parameters can potentially improve the yield, size, and fluorescence properties of the carbon dots, leading to enhanced performance in superhydrophobic coatings and fingerprint detection.

2. Characterization of carbon dot properties: In-depth characterization techniques can be employed to gain a deeper understanding of the physical and chemical properties of the synthesized carbon dots. Techniques such as transmission electron microscopy (TEM), atomic force microscopy (AFM), X-ray diffraction (XRD), and spectroscopic analysis can provide insights into the size, morphology, crystallinity, and surface chemistry of the carbon dots. This information can help correlate the structure-property relationships and further optimize their performance.

3. Functionalization of carbon dots: Exploring different surface functionalization strategies for the carbon dots can expand their range of applications. Surface modifications can involve introducing specific functional groups or coatings to enhance their compatibility, stability, and performance in superhydrophobic coatings and fingerprint detection. This can enable tailoring the carbon dots for targeted applications, such as improved adhesion, increased resistance to environmental factors, or specific interactions with fingerprint residues.

4. Investigation of coating performance: Conducting thorough evaluations of the superhydrophobic coatings incorporating the

fluorescent hydrophobic carbon dots is crucial. Assessments can include testing the durability, water repellency, chemical resistance, and mechanical properties of the coatings. Additionally, studying their self-cleaning ability, long-term stability, and resistance to various environmental conditions can provide insights into their practical applications.

5. Advancements in fingerprint detection: Further exploration of the application of fluorescent hydrophobic carbon dots in fingerprint detection can involve optimizing the detection sensitivity, selectivity, and imaging resolution. Integration with advanced imaging techniques, such as fluorescence microscopy or spectroscopy, can enhance the visualization and identification of fingerprints, leading to improved forensic analysis and crime scene investigations.

6. Expansion of applications: Investigating the potential applications beyond superhydrophobic coatings and fingerprint detection is another avenue for future research. The unique properties of fluorescent hydrophobic carbon dots may find utility in other areas such as sensors, bioimaging, optoelectronics, or drug delivery systems. Exploring these diverse applications can unlock new possibilities and contribute to the development of innovative technologies.

Overall, future research can focus on optimizing the synthesis process, understanding the properties of the carbon dots, exploring surface functionalization, evaluating coating performance, advancing fingerprint detection techniques, and expanding the range of applications for fluorescent hydrophobic carbon dots synthesized through solvothermal processes.

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