

**“SYNTHESIS AND APPLICATION OF ZIRCONIUM DOPED ZINC OXIDE
REDUCED GRAPHENE OXIDE IN SKINCARE”**

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology degree in Chemical Engineering

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**DEPARTMENT OF CHEMICAL ENGINEERING
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SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

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BONAFIDE CERTIFICATE

This is to certify that this project report is a bonafide work of G.DIVYANJALI and SNEHA.S who carried out the project entitled "SYNTHESIS AND APPLICATION OF ZIRCONIUM DOPED ZINC OXIDE REDUCED GRAPHENE OXIDE IN SKIN CARE" under my supervision from NOV 2023 to APR 2023.

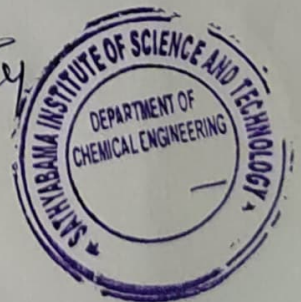
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DECLARATION

We **SNEHA S (40190026)** and **G DIVYANJALI (40190008)** hereby declare that the project Report entitled "**SYNTHESIS AND APPLICATION OF ZIRCONIUM DOPED ZINC OXIDE REDUCED GRAPHENE OXIDE IN SKINCARE**" done by us under the guidance of **Dr. SATHISH.S,M.E.,PH.D.**, at **SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY** is submitted in partial fulfillment of the requirements for the award of **BACHELOR OF TECHNOLOGY DEGREE IN CHEMICAL ENGINEERING**.

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ABSTRACT

This study investigates the biocompatibility of bimetallic reduced graphene oxide (rGO)-based functional nanomaterials. rGO doped with zinc oxide and zirconium dioxide (rGO-ZnO-ZrO₂). The purpose of this effort is to develop a serum that uses the emerging nanomaterial as an active element in skincare. This article describes a method for producing bimetallic reduced graphene oxide using modified Hummers processes. The reduction of GO to rGO was accomplished with the help of L-ascorbic acid. The characteristics of both rGO and Zn Zr/rGO were thoroughly studied utilizing X-ray diffraction (XRD), scanning electron microscopy (FE-SEM), energy dispersive X-ray analysis (EDX), Fourier-transform infrared spectroscopy (FTIR), Brunauer-Emmett-Teller (BET) surface area analysis, and Barrett-Joyner-Halenda (BJH) pore size distribution analysis. And evaluating the graphite serum by its chemical and physical properties. The primary purpose of this effort is to explore the potential of bimetallic rGO-based nanomaterials as active elements in skincare formulations, and to pave the way for the development of innovative skincare products that leverage the unique properties. This research contributes to advancing the knowledge and application of nanotechnology in skincare, offering new opportunities for the formulation of effective and sustainable skincare solutions.

KEYWORDS: Reduced Graphene Oxide, Zirconium doped Zinc Oxide Reduced Graphene Oxide, rGO serum

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LIST OF ABBREVIATIONS

FE-SEM	Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
EDAX	Energy Dispersive X-Ray Analysis
XRD	X- Ray Diffraction
BJH	Barrett-Joyner-HalendaPore Size Distribution Analysis
BET	Brunauer-Emmett-Teller Surface Analysis
GO	Graphene Oxide
rGO	Reduced Graphene Oxide
Zr ZnO/rGO	Zirconium doped Zinc Oxide Reduced Graphene Oxide

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CHAPTER 1

INTRODUCTION

The investigation of novel materials for biomedical applications has attracted a lot of attention lately, especially in the areas of therapeutic interventions, drug delivery, and diagnostics. Reduced graphene oxide is one of these compounds that stands out as a versatile material with remarkable adsorption capabilities. Charcoal, which has long been used in air filtration, water purification, and poison countermeasures, is now being used in the biomedical industry because of its capacity to adsorb infections, poisons, and other organic molecules.

Given this, creating rGO serum seems like a fresh and exciting approach that combines the benefits of reduced graphene oxide with the adaptability of serum-based therapies. Reduced graphene oxide particles are either suspended in or incorporated into rGO serum, a novel formulation. Reduced graphene oxide particles are either suspended in or incorporated into a serum matrix in rGO serum, a novel formulation that provides a platform for specific biological functions and customized adsorption capacities. The purpose of this work is to investigate the characterisation methods, production processes, and possible uses of rGO serum in biomedicine. We want to provide a thorough understanding of this developing material by exploring the nuances of its production process, clarifying its physicochemical features, and evaluating its performance. A number of factors must be taken into account when creating rGO serum, such as choosing suitable sources of charcoal, perfecting integration or suspension methods, and adjusting serum composition to fulfill the required functions.

Additionally, the characterisation of rGO serum requires the use of a number of analytical instruments, including surface area analysis, Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM), to clarify its adsorption capabilities, surface characteristics, and structural characteristic.

This study examines the various uses of rGO serum in biomedicine in addition to its manufacture and characterization. rGO serum has the potential to be used as a medication delivery vehicle and is useful in detoxification therapy and diagnostic assays. It can be used to address a wide range of biomedical issues. Its variable adsorption capabilities and compatibility with biological systems further make it a desirable candidate for targeted therapeutic interventions.

All things considered, the production of rGO serum is an intriguing example of the blending of material science and biology, providing a flexible platform with a broad range of uses. By expanding our knowledge of its production processes, characteristics, and biomedical features, we open doors for the creation of novel approaches in biomedical research, diagnostics, and medication delivery. Our goal in publishing this study is to encourage more investigation and research in this emerging subject, which will ultimately lead to improvements in biotechnology healthcare. In order to create reduced graphene oxide (rGO), graphene oxide (GO) from natural graphite is usually prepared using oxidation techniques like the Hummers' method. To do this, strong oxidizing chemicals are applied to graphite in order to introduce functional groups that contain oxygen. The reduction of GO to rGO is then accomplished by dispersing GO in an appropriate solvent and adding a reducing agent, such as ascorbic acid or hydrazine. Applying heat or using ultrasonication speeds up the reduction process, which removes oxygen functions and sp^2 hybridization recovery. After that, all remaining compounds and impurities are removed from the resultant rGO through purification.

Zn-Zr doping of rGO entails the surface implantation of zirconium-doped zinc oxide (ZnO) nanoparticles. This is accomplished by dissolving zinc and zirconium salts in a solvent to create a precursor solution. The required doping concentration determines the molar ratio of zinc to zirconium. After mixing the ZnO precursor solution with the rGO suspension, the ZnO nanoparticles are encouraged to develop on the rGO surface using hydrothermal or solvothermal treatment.

Through the introduction of zirconium doping, this technique improves the electrical, optical, and structural features of rGO for a range of applications.

The synthesized rGO is characterized by evaluating its structural, morphological, and chemical properties using a range of analytical methods. X-ray diffraction (XRD) for crystalline structure analysis, Raman spectroscopy for assessing structural defects and reduction in degree, scanning electron microscopy (SEM) or transmission electron microscopy (TEM) for morphological analysis, and Fourier-transform infrared spectroscopy (FTIR) for identifying functional groups are examples of common characterization techniques.

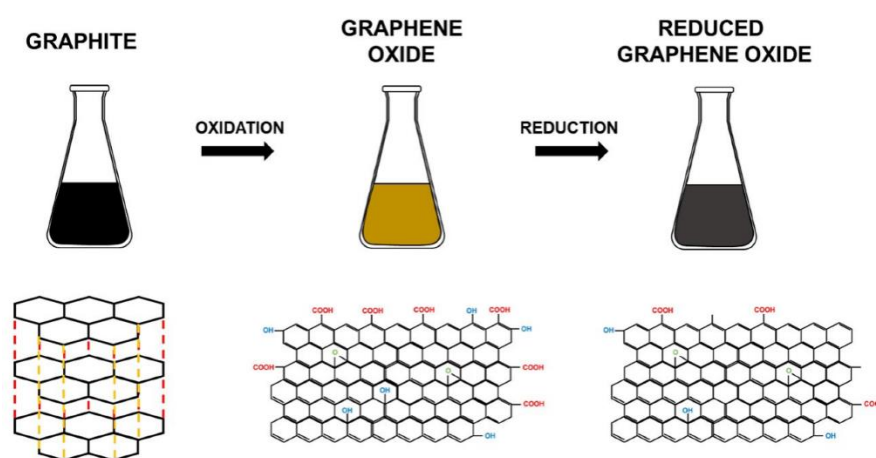


Fig. 1.1: General representation of reducing GO to rGO

These characterisation methods offer important information about the composition and quality of the produced ZrZnO/rGO. In order to create the face serum using an rGO basis, the right ingredients, including water, humectants, emollients, and active compounds, must be chosen to create a proper formulation. The previously produced rGO/ZnO nanocomposite is distributed throughout the serum basis to provide stability and uniform distribution. The desired skincare features determine the incorporation of additional active components like antioxidants, moisturizers, peptides, or botanical extracts. When necessary, the formulation's pH and viscosity are changed to maximize product performance and user satisfaction.

In order to assess the face serum's effectiveness, stability, and safety, a thorough analysis is performed at the end. In vitro and/or in vivo models are used for skin compatibility and irritant testing, while stability studies are carried out under various settings to evaluate shelf life. Physical characteristics including color, smell, and viscosity are tracked over time, and methods are used to measure the amount of active substances. Consumer trials collect input from human participants to evaluate the effectiveness and user experience, guaranteeing that cosmetic goods meet safety regulations and legal requirements. Reduced graphene oxide (rGO) is being synthesized for more than just material production; the goal is to raise awareness about the potential of 2D nanomaterials and skincare uses.

rGO synthesis provides a versatile method that supports this goal. First off, the procedure has inexpensive instrumentation, making it accessible to both practitioners and researchers. Because of its cost, the field of nanoparticles can be extensively explored and experimented with, leading to innovation and discovery. Large product outputs from the procedure also guarantee a steady supply for skincare formulation testing.

Moreover, rGO synthesis's eco-friendliness emphasizes its importance in today's sustainability initiatives. Green chemistry principles are applied in rGO synthesis to minimize environmental impact and to be in line with the increasing worldwide emphasis on sustainable practices. Furthermore, rGO can be produced using the synthesis approach in a variety of morphological forms and sizes, providing flexibility for use in skincare applications. Because of its versatility, researchers can customize rGO characteristics to meet certain skincare requirements while maximizing effectiveness and performance.

The fact that rGO synthesis doesn't require pricy catalysts or hazardous surfactants is another noteworthy benefit. This lowers production costs while guaranteeing the creation of high-quality products devoid of potentially dangerous impurities.

GO synthesis makes it easier to produce skincare formulas that satisfy demanding quality requirements and customer expectations by putting safety and product integrity first.

Using zinc-zirconium (Zn-Zr) doping to synthesize bimetallic reduced graphene oxide (rGO) offers a viable way to improve the characteristics of graphene-based materials used in skincare products. Graphene oxide (GO) is reduced to reduced graphene oxide (rGO) by means of ascorbic acid, which is selected due to its economical and eco-friendly characteristics. As an effective reducing agent, ascorbic acid helps to remove oxygen-containing functional groups from graphene oxide (GO) with the least amount of negative environmental effects.

This environmentally friendly method fits nicely with the growing focus on sustainability in the synthesis of materials. There are several possible advantages to adding rGO to skincare formulas. Because of its distinctive structural characteristics, such as strong electrical conductivity and surface area, rGO shows great promise for improving the effectiveness of skincare solutions. The resulting bimetallic rGO composite can further improve its antibacterial activity, UV protection, and antioxidant qualities by doping it with Zn-Zr. These improvements are especially beneficial for skincare, as it's critical to preserve skin health and shield it from outside stresses.

The effectiveness and efficacy of skincare products can be enhanced by including rGO. For instance, increased skin hydration, elasticity, and attractiveness may result from rGO's capacity to improve skin barrier function and stimulate collagen formation. Its antibacterial qualities can also aid in the fight against bacteria and other pathogens that cause acne, promoting healthier, cleaner skin. Additionally, Zn-Zr dopants may offer other advantages including improved

Antioxidant activity and UV protection also support the health and protection of skin. All things considered, creating bimetallic rGO doped with Zn-Zr is a viable method of incorporating cutting-edge nanomaterials into skincare formulas. Researchers and product developers can take advantage of the special qualities

of reduced graphene oxide (rGO) to build cutting-edge skincare solutions that improve overall skin health, performance, and protection. This can be achieved by utilizing the economical and ecologically friendly reduction. The name "serum," which refers to a highly concentrated product that is frequently used in cosmetology, comes from its professional use. Serums are 10 times more organic than ordinary creams, which helps them address cosmetic issues quickly and effectively. The need for cosmetic items has surged due to the rising worldwide cost of living, hence situating the cosmetics business.

As more people strive to stay young and appealing, cosmetics have become more and more valuable. Whether in the form of a gel, light lotion, or moisturizer, a skin serum has the capacity to deeply permeate the skin and supply the skin with active chemicals. It can reduce pores, increase moisture content, and improve the firmness and texture of the skin. Antioxidants, chemicals derived from cells, and substances resembling skin are essential for preserving skin health in all forms. Richer moisturizing creams are advised for dry to extremely dry skin, while serums and light lotions work best for normal to dry skin. Gel formulations and liquids are ideal for oily or combo skin. The essential components for skin health are the same in all products, regardless of differences in skin texture. The largest and most defensive organ in the body is the skin, can become dry as a result of a number of things, including allergies, debris, UV rays, and leftover makeup.

Because serum has small molecules, it may be applied directly to specific skincare concerns like uneven skin tone or age indications. Applying serum after cleansing but before moisturizing allows for this.

Cosmeceuticals are skincare products that combine cosmetic and medical characteristics. They are a blend of cosmetics and pharmaceuticals. As defined by the Health Sciences Authorities' Guide to the Control of Cosmetic Products (Revised 2014), a cosmetic product is anything that is meant to be applied to the skin, hair, nails, lips, eyes, teeth, and external genitalia in order to clean, perfume, change appearance, control odor, or provide protection. Usually, these products fall into one of five primary categories. Enhancing the skin's barrier function, an

emulsion-based face serum serves as a moisturizer and a delivery system for powerful skincare products. Emulsions combine two phases that don't seem to go together, like water and oil, by stabilizing the mixture with emulsifiers. This makes it possible to produce a stable product that successfully blends the advantages of the two stages.

An emulsion-based serum's primary benefit is its capacity to introduce active ingredients deeply into the skin's tissues. This is important since the skin's natural barrier makes it difficult for elements in cosmetics to penetrate deeply. Through the use of water and oil in an emulsion, the serum works to increase the probability that these potent active ingredients will reach the dermis and begin to work. Most successfully. Additionally, the emulsions' natural moisturizing qualities reinforce the skin's barrier function, promoting the general resilience and health of the skin. To ensure the serum's efficacy, safety, and quality in skincare compositions, each test that is performed on it is vital. By detecting and measuring any microbiological contamination, this analysis guarantees that the serum is free of dangerous pathogens that could infect skin or produce unfavorable reactions when applied topically.

Studies on the serum's stability are carried out to evaluate its capacity to retain quality overtime the physical properties and assessment of the rGO serum concentrate on identifying any alterations or distinctiv First and foremost, the spreadability test is important since it assesses how readily the serum can be administered and dispersed throughout the surface of the skin. Uniform coverage and absorption are ensured by a serum with ideal spreadability, which improves user experience and maximizes the administration of active ingredients, determining the reduced graphene oxide (rGO) serum's microbiological safety requires a microbial analyse features related to the presence of reduced graphene oxide. This assessment offers important insights into how rGO is incorporated into the serum matrix, guaranteeing its efficacy and suitability for use in skincare products. stability and compatibility. It affects the effectiveness of the active components in the serum as well as the skin barrier function and irritation

potential. To maximize the serum's benefits and reduce the chance of skin irritation, make sure its pH is within the physiological range of the skin.

Lastly, the viscosity measurement contributes to our understanding of the consistency and texture of the serum, which influences its spreadability, application ease, and skin absorption. Formulators can obtain the required product attributes and ensure optimal user experience and efficacy by assessing viscosity and adjusting the formulation accordingly.

All things considered, the results of these serum tests collectively offer thorough insights into the product's stability, safety, and effectiveness, guaranteeing the creation of superior skincare products that satisfy both legal and consumer requirements. A noteworthy advancement in skincare technology is the development of arGO (reduced graphene oxide) serum. Through the utilization of rGO's extraordinary characteristics, such as its large surface area, high conductivity, and biocompatibility, we have the potential to completely transform skincare formulas. With these qualities at our disposal, we want to develop innovative skincare products that successfully address a wide range of cosmetic issues.

Our mission is to fully realize the potential of rGO-based serums with a rigorous testing protocol and meticulous experimentation process. Our attempts to bring in a new era of skincare innovation are based on this commitment to scientific study and quality assurance. Our ultimate goal is to launch next-generation skincare products that not only offer unmatched effectiveness while giving sustainability of the environment and safety equal priority. By utilizing rGO, we hope to raise the bar for the skincare sector and enable people all around the world to have healthier, more beautiful skin.

The groundwork for our novel skincare serum is laid during the first part of our formulation process, where we carefully prepare the oil and water phases, to aid in emulsification. We provide the foundation for future investigations and

understandings of the serum's functionality and effectiveness by thoroughly evaluating the serum's globule size and physical characteristics

When graphene oxide is heated or chemically treated, reduced graphene oxide (rGO) is produced. This is a noteworthy development in the field of material science. This process uses reducing agents such as sodium borohydride, L-ascorbic acid, hydrazine, and hydrazine hydrate to occur between graphite layers and oxidized graphene oxide. Notably, recent studies have investigated the application of plant extracts for rGO synthesis, taking advantage of their accessibility and friendliness.

These extracts help to produce nanoparticles by acting as capping and reducing agents. Furthermore, rGO's functional groups allow for a variety of chemical interactions with biomolecules, including enzymes, proteins, peptides, and DNA. Biomolecules are being used more often in rGO synthesis due to their low labor costs, biocompatibility, and potential uses in a variety of industries, including medicine. Additionally, rGO's low toxicity increases its potential. Its high surface area enhances its effectiveness when used therapeutically.

Through a variety of methods, composites of rGO with metals and metal oxides have demonstrated great potential in improving supercapacitor performance. In order to create rGO nanocomposites embellished with Ag and MnO₂ nanoparticles, for example, Ma et al. presented a two-step synthesis process that produced uniformly distributed structures favorable to improved performance in energy storage applications. This process demonstrated the adaptability and potential of rGO in composite materials by first developing MnO₂ nanostructures on GO sheets in situ, then reducing GO and Ag⁺ to create Ag/MnO₂/rGO composites.

In many scientific and industrial applications, reduced graphene oxide (rGO), a crucial substance that is produced by reducing graphene oxide through chemical or thermal methods, fills a large role. The reduction process usually occurs between graphite layers and oxidized graphene oxide, using a variety of agents

including sodium borohydride, l-ascorbic acid, hydrazine, and hydrazine hydrate. But because plant extracts are readily available and environmentally benign, efforts in this area have recently shifted to using them. The addition of biomolecules not only speeds up the reduction process but also gives the final rGO biocompatible characteristics that make it suitable for a wide range of biomedical uses. Because of its low labor intensity and cost-effectiveness, this combination of reduced graphene oxide and biological entities has attracted interest as a promising. Biotechnology and materials science avenue. Furthermore, rGO's low toxicity by nature enhances its therapeutic potential and makes it easier to use in biomedical domains.

In addition to being biocompatible, rGO's large surface area has led to its incorporation into composite materials, increasing its usefulness in a variety of applications, including catalysis and energy storage. Recent studies, for example, have concentrated on creating rGO-based nanocomposites using complex synthesis techniques, producing materials with improved qualities for supercapacitor applications. Silver (Ag) and manganese dioxide (MnO₂) nanoparticles are evenly distributed onto rGO sheets in a two-step procedure that synthesizes a rGO nanocomposite.

This example illustrates the possibility for customized fabrication techniques to fine-tune the attributes of rGO composites. This expanding field of study highlights the adaptability and expanding potential of rGO in tackling current issues in a variety of multidisciplinary fields, such as energy storage friendliness. Regarding and biomedicine, and opening the door for creative technical developments. In recognition of its special qualities and adaptability, reduced graphene oxide (rGO) has attracted a lot of attention lately for use in a variety of applications. The synthesis, characterisation, and use of rGO in face serum compositions are the main topics of this research. rGO, which is produced by thermally or chemically treating graphene oxide (GO), is a desirable option for biomedical applications because to its exceptional electrical conductivity, high surface area, and biocompatibility.

In order to create rGO, oxygen-containing functional groups in GO must be reduced. This restores the sp^2 carbon network and creates a highly conductive material. For this reason, reducing agents including sodium borohydride, L-ascorbic acid, hydrazine, and hydrazine hydrate are frequently utilized. Numerous techniques, including chemical For the synthesis of rGO, reduction, thermal reduction, and green synthesis utilizing plant extracts have all been investigated; each has special benefits in terms of scalability, affordability, and environmental friendliness.

Regarding face serum compositions, rGO has a number of possible advantages, such as improving the absorption of active compounds into the skin, encouraging the production of collagen, and possessing antioxidant qualities.

Furthermore, the large surface area of rGO offers plenty of room for the loading and long-term release of bioactive substances, enhancing the stability and effectiveness of face serums.

we offer a thorough investigation into the environmentally friendly synthesis of rGO employing plant extracts as reducing agents. Using cutting-edge analytical methods, we describe the synthesized rGO and assess its potential use in face serum compositions. Additionally, we look into The findings of in vitro and in vivo studies on the impact of rGO incorporation on the physicochemical characteristics and functionality of face serums. The overall goal of this research is to use the special qualities of rGO to help produce novel and efficient skincare solutions.

In the realm of skincare, innovations continually emerge to address various skin concerns and deliver targeted benefits. Among these innovations, charcoal serum stands out as a potent and versatile solution for achieving healthier, clearer, and more radiant skin.



Fig. 1.2: Skin with freckles and dark spots

Graphite serum represents a groundbreaking approach to skincare, harnessing the purifying and detoxifying properties of graphite to deeply cleanse and rejuvenate the skin. As a skincare product applied after cleansing and before moisturizing, graphite serum is formulated to deliver concentrated amounts of active ingredients to the skin, providing rapid and effective results.

Key Features of graphite Serum:

1. Purifying and Detoxifying: graphite acts as a magnet for impurities, toxins, and excess oil, drawing them out from deep within the pores and leaving the skin thoroughly cleansed and refreshed.
2. Deep Hydration: Despite its powerful detoxifying properties, graphite serum also provides deep hydration to the skin, helping to maintain moisture balance and prevent dryness.
3. Skin Brightening: graphite serum may contain brightening agents that help to even out skin tone, reduce the appearance of dark spots and hyperpigmentation, and promote a more radiant complexion.

4. Anti-Acne Properties: graphite serum can be beneficial for those prone to acne or blemishes, as it helps to unclog pores, reduce inflammation, and prevent future breakouts.
5. Anti-Aging Benefits: Some graphite serums may also contain anti-aging ingredients such as antioxidants and peptides, which help to combat the signs of aging, including fine lines, wrinkles, and loss of elasticity.

In conclusion, the synthesis of reduced graphene oxide is a driving force behind the growing knowledge and comprehension of two-dimensional nanomaterials and their possible uses in skincare.



Fig .1.3: rGO face serum

Because of its inexpensive instrumentation, rGO synthesis opens the door for cutting-edge skincare products that are sustainable and effective thanks to its high product yield, eco-friendly manufacturing technique, and capacity to create high-quality, customization nanomaterials. The unique qualities and formulation of the RGO face

serum offer a promising range of skincare advantages. This serum is an adaptable way to help with different skin issues and support general skin health. RGO's anti-aging qualities are one of its main purposes; being high in antioxidants, it fights free radicals and lessens the appearance of wrinkles, fine lines, and age spots. Its superior moisture-trapping ability further adds to skin hydration, making it a great option for anyone with dry or dehydrated skin. Moreover, RGO has skin-brightening qualities that improve complexion radiance and even out skin tone. Because of its anti-inflammatory properties, it's very useful for relieving discomfort, calming inflamed skin, and lowering redness. Furthermore, RGO's antibacterial qualities make it useful in the fight against germs that cause acne, which makes it an important part of acne treatment plans. All things considered, adding RGO face serum to regular skincare regimens can result in healthier, more radiant skin by addressing a variety of issues like aging indications, dehydration, and imperfections.

CHAPTER 2

LITERATURE SURVEY

- **Kanchan Chande¹ , Sunidhi Dessai² , Santosh Bhutekar³ , Md Talat Choudhary⁴ , Kamlesh Choudhary⁵ , Shriram Bairagi⁶ , Smita Takarkhede** have published a scientific article ; This essay focuses on the traditional application of herbal medicine, specifically chrysanthemum flower, for medicinal and skincare purposes. This practice is common not only in India but also in other parts of the world. Given that chrysanthemum flowers are rich in vitamins, flavonoids, carotenoids, anthocyanins, and essential oils—all of which have multiple advantages for skin health—the flower's inclusion in skincare products makes sense. The discovery of chrysanthemum's antibacterial and anti-inflammatory qualities highlights the plant's potential for treating common skin conditions like acne and inflammation brought on by UV radiation & photo damage.

In addition, the creation of a face serum with glycerin, vitamin E, and chrysanthemum is a good skincare strategy. Concentrated formulations of face serums are well-known for enabling deeper penetration into the skin layers, optimizing the active ingredient delivery. Due to its antioxidant qualities, vitamin E is included, which increases the serum's ability to fight environmental damage and aging of the skin. Though traditional herbal medicines have been used for many years, it's important to remember that their safety and efficacy need to be confirmed by rigorous research and clinical trials. Additionally, a number of variables, such as the formulation, the concentration of active substances, and the type of skin, affect how effective skincare products are.

This research concludes by highlighting the potential of chrysanthemum flower in cosmetics formulations and emphasizing the value of investigating natural components for their possible medicinal uses. Additional study in this field may result in the creation of secure and efficient.

- **Sanjana Patil¹ , Akshada Deshmukh² , Dr. S.V Patil³ Prajakta Patil⁴ , Riya Patil⁵ , Pratiksha Patil⁶ , Shivtej Patil⁷ , Sayali Hogade** have published the paper The creation of a serum with a polyherbal extract made of coconut oil, almond oil, glycerin, and aloe vera gel is covered in the paper. The purpose of adding these chemicals to the serum recipe is to take advantage of each one's unique skincare benefits. The ability of aloe vera extract, which is well-known for increasing collagen, to reduce aging symptoms including wrinkles and fine lines is underlined. Liquorice, which contains glycyrrhizin, is also known for its antioxidant qualities, which can help shield the skin from UV rays and free radical damage, thereby delaying the aging process. The serum's high concentration of active chemicals, deep penetration into the skin's layers, rapid absorption, and non-greasy finish are all said to contribute to its efficacy. The application The antioxidant benefits of coconut oil and its ability to prevent premature skin aging are also highlighted. All things considered, the study offers insightful information about how to create a serum that is enhanced with organic components that are well-known for their positive effects on aging and skin health. Nevertheless, additional investigation and clinical trials could be required to confirm the effectiveness and safety of the designed serum for use in skincare applications.
- **Shan Sasidharan^{1*}, Pyarry Joseph ², Junise** have published a paper The formulation of a fairness serum using polyherbal extracts is presented in the study, with special attention to the serum's high concentration of active ingredients and quick skin absorption. Because of its noticeable effects, the serum promises to give not only rapid cosmetic results but also psychological happiness after treatment. Known for their ability to enhance fairness, a blend of active natural components, including Glycyrrhiza glabra, Crocus sativus, rice bran, and olive oil, are included in the formulation. The serum's capacity to permeate the skin's color cells and epidermis, improving skin tone and resulting in a fairer complexion, is emphasized. To further guarantee the formulation's safety and effectiveness, extensive analyses, including physicochemical and biological assessments, were conducted. The lack of heavy metals, high spreadability, and non-sensitizing qualities of the substance made it appropriate for employ. All things considered, the study provides insightful information about the creation of a fairness serum using polyherbal extracts, which may be helpful for

skincare lovers looking to achieve noticeable and durable improvements in skin tone and texture. The efficacy and safety profile of the serum may need to be confirmed through additional research and clinical studies before it can be widely used in skincare applications.

- **Tejal Shirish Shejul1* Prof. Kiran Kudale1*** has published a paper The study discusses the rising demand for skincare treatments and products and stresses how crucial it is to uphold appropriate look and adhere to modern beauty standards. It emphasizes the value of adding serums to skincare regimens and points out how adaptable they are in terms of meeting the needs of many skin types and issues. In the literature study, the advantages of utilizing serums with particular components designed to meet the demands of each different skin type—like brightening and healing qualities—are highlighted. It also addresses the possibility of using concentrated vitamin and acid serums to treat skin aging issues with encouraging outcomes. All things considered, the research offers insightful information about the function of serums in skincare routines and the effectiveness of specialty formulations in reaching targeted skincare results. But more investigation and clinical trials are needed. May be required to confirm whether serums are beneficial in treating particular skin issues and delaying the aging process.
- **Smriti Ojha, surabhi sinha, swadhapriya das chaudhuri, hina chadha** have published a paper The article tackles the prevalent issues of wrinkles on the face and skin aging due to UV exposure and photodamage, highlighting the deficiency of efficacious methods to counteract the aging process. By creating a highly concentrated face serum with glycerin, honeybee venom, and aloe vera, it presents a fresh technique. Aloevera is emphasized for its many uses in skincare products because of its quick absorption, capacity to reach deeper skin layers, and medicinal qualities, which include antibacterial, anti-inflammatory, and wound-healing capabilities. Moreover, the serum composition gains a novel dimension from the incorporation of honeybee venom, which is well-known for its purported capacity to enhance blood flow and collagen formation. The assessment of the serum's physicochemical properties is covered in further detail in the publication, including pH, globule size, uniformity, and stability study

findings showing no appreciable variations in globule size or visual appearance over time. In summary, the study offers a potentially fruitful path for innovative skincare by utilizing the combined benefits of organic components to tackle prevalent skin issues and possibly postpone the aging process. In order to confirm the effectiveness and safety of this innovative face serum composition for wider skincare uses, more investigation and clinical trials could be necessary.

- **T.S. Sreeprasad, Shihabudheen M. Maliyekkal, K.P.Lisha, T.Pradeep** have published a paper In order to generate reduced graphene oxide (RGO)–metal/metal oxide composites and use them in water purification, this research provides a simple and flexible synthetic method. The composites were created from precursor ions by utilizing the intrinsic reduction capability of RGO, which produced materials with improved pollution removal capabilities. The produced composites were characterized using a variety of spectroscopic and microscopic techniques, which revealed a redox-like reaction process between RGO and the metal precursor that led to the anchoring of metal nanoparticles onto the carbon sheets. In comparison to pure RGO and graphene oxide (GO), RGO–metal/metal oxide composites, specifically RGO–MnO₂ and RGO–Ag, showed notably greater distribution coefficients (K_d) for scavenging metal ions, specifically Hg(II). The creation of a technique to render the composites immobile on rivers Their potential for field applications is further increased by sand that uses chitosan as a binder. Overall, by proposing effective adsorbent materials with useful implications for environmental remediation, this research advances water purification technologies. The scalability and long-term effectiveness of these composites in actual water treatment settings may be investigated in more research.
- **B.Sravani, P.Raghavendra, Y.Chandrashekar , Y.Veera Manohara reddy** have published a paper on in order to improve the kinetics of electrochemical reactions—particularly those that are pertinent to fuel cell applications—this work tackles the crucial necessity for bimetallic nanoparticles evenly distributed over conductive carbon substrates. The goal of the project is to apply a straightforward one-pot chemical reduction approach to create bimetallic platinum-cobalt and platinum-nickel nanoparticles on reduced graphene oxide (rGO) substrates with a high degree of

uniform dispersion. Notably, the high acidity of the graphene oxide produced during chemical exfoliation was neutralized by pomegranate peel extract, which made it easier to synthesize well-dispersed Pt₃Ni/rGO and Pt₃Co/rGO nanoparticles.

The effectiveness of the synthesized catalysts in the electrocatalytic oxygen reduction process (ORR) in basic and acidic environments was assessed. According to the findings, Pt₃Ni/rGO nanocatalysts show noticeably greater ORR activity descriptors, such as area- and mass-specific current densities. In contrast, Pt₃Co/rGO exhibits the highest ORR activity in basic medium when compared to the other evaluated catalysts.

The study highlights how various mono, bi, and ternary metallic nanoparticles can be generated with great uniformity on carbon-based substrates using the simple fabrication approach that has been provided, opening the door to the production of more sophisticated electrocatalysts. All things considered, this study offers insightful information about the synthesis and design of effective electrocatalysts for a range of energy conversion and storage applications. It seems promising to investigate this fabrication approach further in order to produce new materials with improved electrochemical performance.

- **Fangfang Ren[†] Huiwen Wang[†] Chunyang Zhai[†] Mingshan Zhu[§] Ruirui Yue[†] Yukou Du[†] Ping Yang[†] Jingkun Xu, and Wensheng Lu** have published a scientific paper. In order to create PtPd/reduced graphene oxide (RGO) catalysts with different Pt/Pd ratios, this article describes a new and environmentally benign approach that does not require the use of stabilizers, surfactants, or external energy. X-ray diffraction (XRD), transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), induced coupled plasma atomic emission spectroscopy (ICP–AES), and electrochemical measurements were among the many techniques used to thoroughly characterize the synthesized catalysts. All metallic nanoparticles (NPs) had clearly defined crystalline structures, according to the HRTEM examination, demonstrating the high caliber of the catalysts that were produced. Crucially, the molar ratio of the Pt and Pd precursors could be readily changed to modify the composition of the Pt–Pd/RGO catalysts,

providing control over their characteristics. Studies involving electrochemistry, such as cyclic voltammetry (CV) the creation of PtPd alloy catalysts that are specifically suited for use in fuel cells, featuring improved catalytic activity and customized compositions. Potential for improvements in the field of electrocatalysis exists due to the clean synthesis approach and the capacity to regulate the catalyst composition. With practical applications in energy conversion technologies, this discovery opens up new opportunities for the creation of effective and environmentally sustainable catalyst materials.

CHAPTER 3

AIM AND SCOPE

AIM

The aim is to synthesize reduced graphene oxide and dope it with zirconium and zinc. Characterization of rGO and bimetallic rGO is done. Development and characterisation of a face serum using Zr doped ZnO/rGO in a skin care product.

SCOPE

The purpose of synthesizing reduced graphene oxide (rGO) for skincare applications, as described, serves several important goals:

- **Improving Awareness about 2D Nanomaterials:** By focusing on rGO, which is a two-dimensional nanomaterial, the synthesis process helps raise awareness about the potential of such materials in skincare. Educating consumers and industry professionals about the benefits and applications of 2D nanomaterials can foster innovation and adoption in skincare product development.
- **Low-Cost Instrumentation and Large Yield:** The use of low-cost instrumentation and achieving a large yield of products make the synthesis process economically viable. This aspect is crucial for scalability and affordability, ensuring that skincare products incorporating rGO can be produced cost-effectively and made accessible to a wider audience.
- **Environment-Friendly Process:** Employing an environmentally friendly synthesis process aligns with sustainability goals and reduces the ecological footprint of skincare product manufacturing. This aspect is increasingly important in the

beauty and personal care industry, where consumers are seeking products that are environmentally conscious and sustainable.

- **Synthesis of Controlled Morphologic Forms:** The ability to synthesize different morphological forms of nanomaterials with controlled size offers versatility in skincare product development. Tailoring the morphology of rGO can optimize its properties for specific skincare applications, enhancing its efficacy and performance.
- **Avoidance of Expensive Catalysts and Harmful Surfactants:** By eliminating the need for expensive catalysts or harmful surfactants in the synthesis process, the quality of the rGO products is maintained while reducing production costs and environmental impact. This ensures that high-quality skincare products can be developed without compromising on safety or efficacy.

CHAPTER 4

MATERIALS AND METHADODOLOGY

The Hummers method is commonly used to prepare graphene oxide (GO) from graphite. To synthesize reduced graphene oxide (rGO) from GO using a modified Hummers method, you'll essentially be reducing the oxygen-containing functional groups in GO to obtain a more graphene-like material. Here's a step-by-step procedure for this synthesis.

4.1 MATERIALS REQUIRED:

1. Graphite powder
2. Sodium Nitrate (NaNO_3)
3. Sulfuric acid (H_2SO_4)
4. Phosphoric acid (H_3PO_4)
5. Potassium permanganate (KMnO_4)
6. Hydrogen Peroxide (H_2O_2)
7. Zinc Oxide (ZnO)
8. Zirconium dioxide (ZrO_2)

4.2 EQUIPMENTS REQUIRED:

- Hot plate magnetic stirrer
- Ultra sonicator
- Hot air oven
- Ice bath

- Filtration setup

4.3 SYNTHESIS OF GRAPHENE OXIDE:

- Initial Mixing: Mix 5 g of graphite flakes with 2 g of sodium nitrate (NaNO_3) in a 1000 mL volumetric flask containing 115 mL of a mixture of sulfuric acid (H_2SO_4) and phosphoric acid (H_3PO_4) in a 9:1 ratio. Keep the flask in an ice bath and stir continuously.
- Oxidation Initiation: Stir the mixture for 1 hour in the ice bath, ensuring a well-mixed suspension. Slowly add 15 g of potassium permanganate (KMnO_4) to the suspension while stirring. Control the rate of addition to maintain a reaction temperature below 15°C .
- Reaction Progress: Remove the ice bath and continue stirring the mixture at 35°C . The color of the mixture will turn pasty brownish-black.
- Dilution and Temperature Increase: Dilute the mixture by adding 100 ml of water, and the reaction temperature will rise rapidly to 98°C with effervescence. The color will change to brown.
- Further Dilution: Continue dilution by adding an additional 200 ml of water while stirring continuously.
- Termination of Reaction: Add 10 ml of hydrogen peroxide (H_2O_2) to terminate the reaction. The appearance of a yellow color indicates the end of the reaction.
- Purification: Rinse the mixture with deionized (DI) water several times to remove impurities.
- Filtration and Drying: Filter the mixture to separate the solid material from the liquid. Dry the obtained graphene oxide (GO) as a powder in a hot air oven.

4.4 SYNTHESIS OF REDUCED GRAPHENE OXIDE:

- Preparation of Mixture: Combine 50 ml of the GO solution with 3 grams of ascorbic acid in a volumetric flask. Ensure the ascorbic acid is well-dissolved in the GO solution.
- Heating and Stirring: Heat the mixture to 70°C and stir the mixture vigorously using a magnetic stirrer. Maintain this condition for 2 hours, allowing the reduction reaction to take place.
- Observation of Color Change: During the reaction, you will notice a significant change in the color of the solution. It will transition from brownish yellow (characteristic of GO) to black, indicating the reduction of GO to rGO.
- Centrifugation: After the 2-hour reaction, centrifuge the mixture at 5000 rpm to separate the solid rGO from the liquid (supernatant). The centrifugation step helps collect the rGO.
- Supernatant Removal: Carefully decant and remove the supernatant, leaving the solid rGO at the bottom of the centrifuge tube.
- Drying: Place the collected rGO sample in a hot air oven and dry it at 60-80°C for several hours to remove any remaining water or solvent. This process yields powdered reduced graphene oxide.

4.5 SYNTHESIS OF Zr DOPED Zn RGO

- Add 1.5 g of ZnO, 1.5 g of ZrO₂, and 4 g of rGO into a 500 ml Erlenmeyer flask.
- Pour 300 ml of distilled water into the flask, creating a mixture of the metal oxide powders (ZnO and ZrO₂) and the rGO.
- Place the Erlenmeyer flask on a magnetic stirrer, using a large rotor, to ensure vigorous mixing of the materials.

- Control the pH value to maintain it at 8.0 during the stirring process. This may involve adding an acid or base as necessary to achieve and maintain the desired pH.
- Continue stirring the mixture vigorously for 1 hour to ensure good dispersion and interaction of the materials.
- At the end of the stirring period, precipitation will be visible in the flask.
- Stop the stirring and remove the rotor.
- Allow the mixture to stand still so that the precipitation settles at the bottom of the flask.
- After settling, the supernatant liquid can be carefully decanted, leaving the precipitate in the flask.
- Wash the precipitate 3-5 times with distilled water to remove any residual reactants or impurities.
- Set up a vacuum filtration system to separate the wet precipitate from the liquid.
- Once the precipitate is separated and collected, vacuum-dry it at 60°C for several hours to remove any remaining water.
- After drying, Zr-doped ZnO/rGO nanocomposites is obtained.

4.6 PREPARATION OF rGO SERUM:

Oil Phase Preparation:

1. Mix together the following ingredients:

- Olive oil
- Sandalwood oil
- Tween 20 (a surfactant used to help disperse oil in water)

- Coconut oil

2. Stir the mixture for 10 minutes to ensure the oils are uniformly blended. This creates your oil phase.

Water Phase Preparation:

In a separate container, combine the following ingredients:

- Aloe vera gel
- Glycerin
- Salicylic acid (often used for its exfoliating properties)
- Vitamin C (a common antioxidant in skincare products)
- A small amount of distilled water
- Add Zr-doped ZnO/rGO (Zirconium-doped Zinc Oxide/reduced Graphene Oxide)

Emulsification:

While mechanically vibrating or stirring (e.g., using a high-speed homogenizer), add the oil phase dropwise into the water phase. Continue to mix the two phases together at 2500 rpm or until a stable oil-in-water emulsion is formed. This means the oil phase (containing the oils and other ingredients) is dispersed as tiny droplets within the water phase.

Incorporation of Graphite:

If graphite is to be incorporated into the serum for its properties, it should be added during the water phase preparation. Ensure proper dispersion of graphite particles in the water phase before emulsification.

Final Steps: Once the emulsion is stable and the desired consistency is achieved, the graphite serum is ready for packaging and use.



Fig.4.1: Stirring of rGO solution in waterbath



Fig.4.2: rGO solution under magnetic stirrer for 12hours



Fig.4.3: Filtered rGO



Fig.4.4: Dried rGO powder



Fig.4.5: Final product of rGO face serum

CHAPTER 5

RESULT AND DISCUSSION

5.1 CHARACTERIZATION

5.1.1 SCANNING ELECTRON MICROSCOPE

Reduced Graphene Oxide:

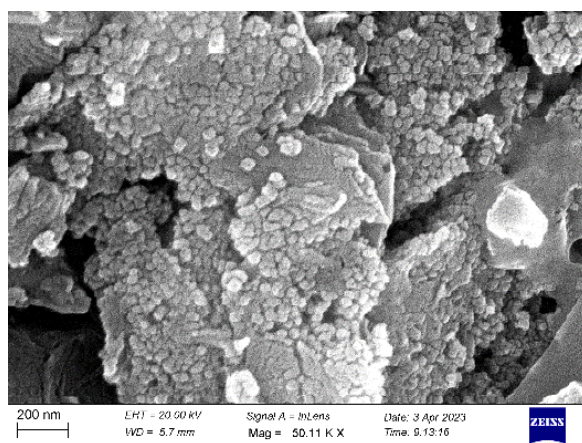


Fig. 5.1: FESEM image of reduced graphene oxide

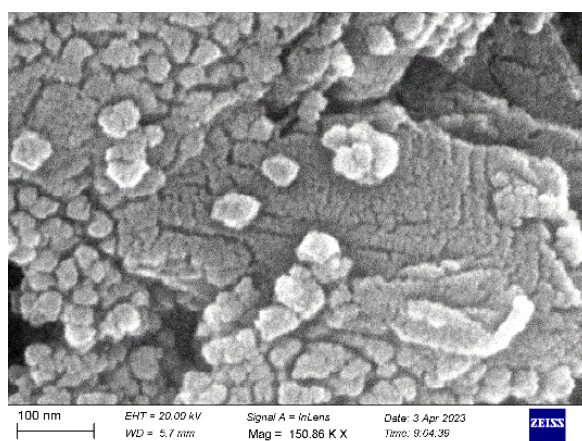


Fig. 5.2: FESEM image of reduced graphene oxide

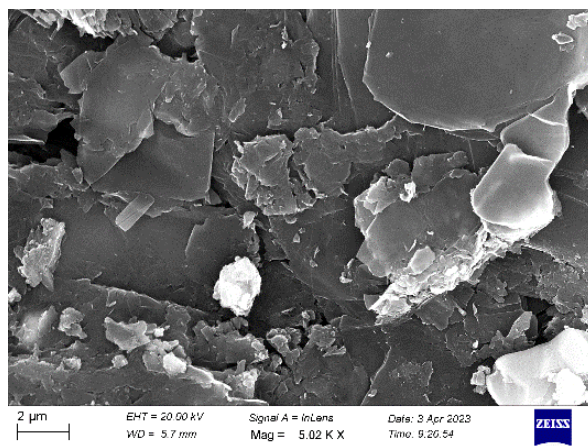


Fig. 5.3: FESEM image of reduced graphene oxide

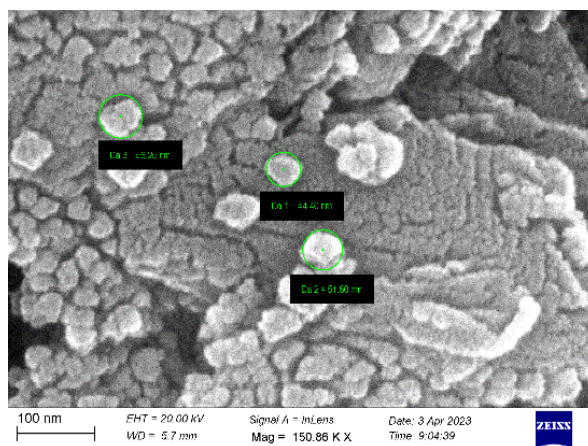


Fig.5.4: FESEM image of reduced graphene oxide

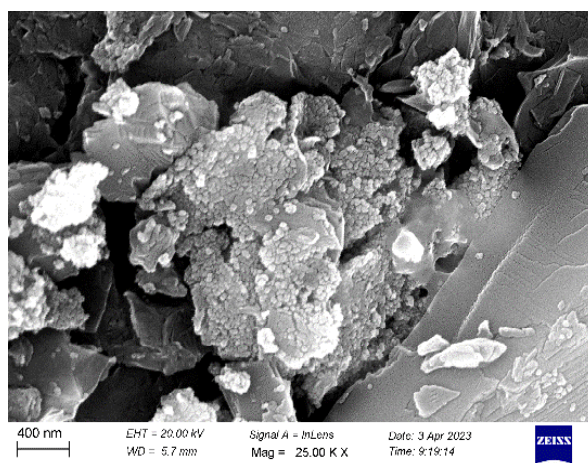


Fig.5.5: FESEM image of reduced graphene oxide

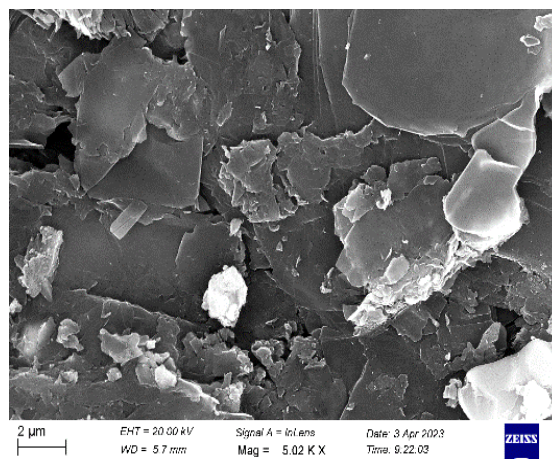


Fig.5.6: FESEM image of reduced graphene oxide

1. Morphology of rGO:

- The SEM micrographs reveal that rGO possesses a two-dimensional sheet-like structure.
- rGO exhibits a multiple lamellar layer structure with distinguishable edges between individual sheets. This indicates the presence of stacked graphene layers.
- The individual rGO sheets appear relatively thick, with distinguishable thickness.
- The thickness of the rGO sheets is attributed to the introduction of oxygen-containing functional groups during the reduction process.
- The edges of the rGO sheets appear thicker due to the concentration of oxygen-containing functional groups at these locations.

2. Stability and Structure:

- The SEM images indicate that the rGO sheets are firmly suspended and do not exhibit bending. This suggests good stability and integrity of the rGO structure.
- The observed stability is crucial for potential applications in skincare, as it ensures the uniform dispersion of rGO within skincare formulations.

3. Particle Diameter:

- The particle diameter of the rGO ranges from 44.04 nm to 56.25 nm, indicating the size distribution of the rGO sheets.

- This particle diameter range provides insight into the size of individual rGO sheets, which can influence their properties and behavior in skincare formulations

Zr doped Zn Reduced Graphene Oxide:

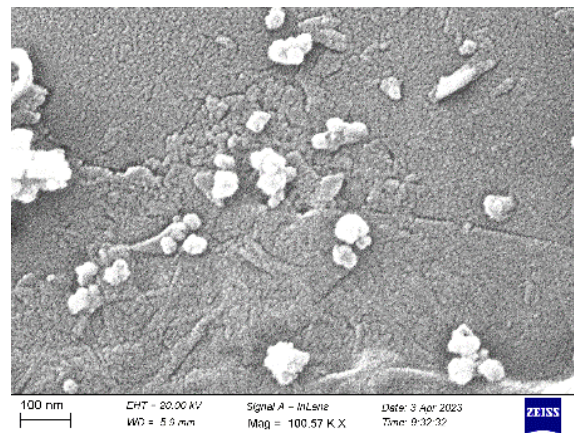


Fig.5.7: FESEM image of Zr ZnO/rGO

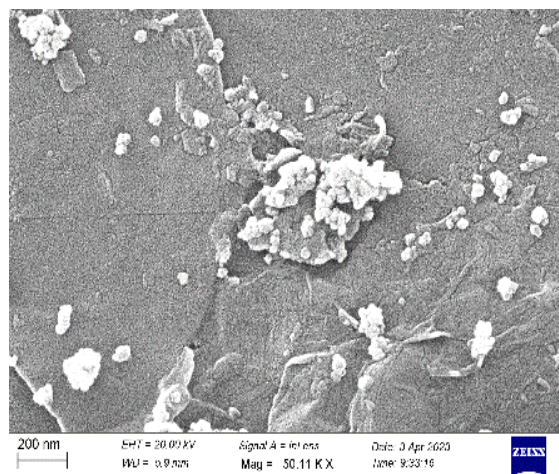


Fig.5.8: FESEM image of Zr ZnO/rGO

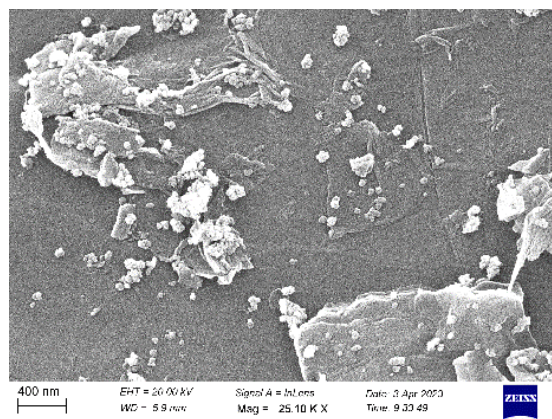


Fig. 5.9: FESEM image of Zr ZnO/rGO

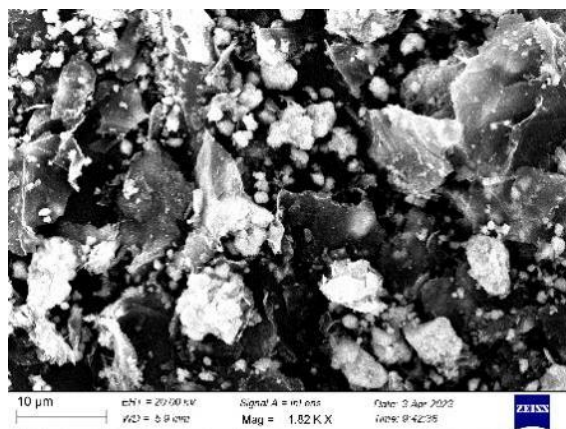


Fig. 5.10: FESEM image of Zr ZnO/rGO

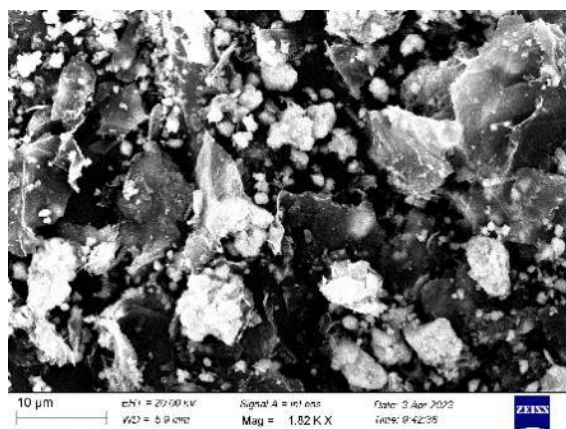


Fig. 5.11: FESEM image of Zr ZnO/rGO

The SEM characterization results for Zirconium-doped ZnO (ZnO/ZrO₂) nanoparticles and their assimilation with reduced graphene oxide (rGO):

1. Surface Morphology:

- The micrographs indicate that the surface morphology of ZnO nanoparticles (NPs) and the incorporation of ZrO₂ doping did not significantly alter the particles' surface.
- This suggests that the doping process did not induce significant changes in the surface structure of ZnO NPs, maintaining their original morphology.

2. Homogeneous Anchoring of Particles on rGO Nanosheets:

- The SEM micrograph of ZnO/ZrO₂ nanocomposites (NCs) on rGO nanosheets reveals that particles were homogeneously anchored on the rGO surface.
- This indicates successful assimilation of ZnO/ZrO₂ NCs with rGO, forming a composite structure where nanoparticles are dispersed and deposited onto the rGO surface.

3. Microscopy Images of Zr-Doped ZnO/rGO:

- The microscopy images of Zr-doped ZnO/rGO, observed at 30K magnification, show a layered planar structure with a wrinkled surface of rGO.
- The rGO structure exhibits a wrinkled surface, which is consistent with previous studies and indicative of its characteristic morphology.
- The particles of ZnO and ZrO₂ are observed to be bound together by stacking on each other, forming clusters or aggregates.

4. Surface Modification and Deposition on rGO:

- Surface modification was performed after the preparation of Zr-doped ZnO/rGO nanomaterials, resulting in the deposition of various sizes of ZnO and ZrO₂ nanoparticles on the rGO surface sheets.
- The deposition of nanoparticles on the rGO surface indicates successful bonding during the pyrolysis process, leading to the formation of Zr-doped ZnO/rGO composite materials.

EDAX(ENERGY DISPERSION X RAY ANALYSIS)

Reduced Graphene Oxide:

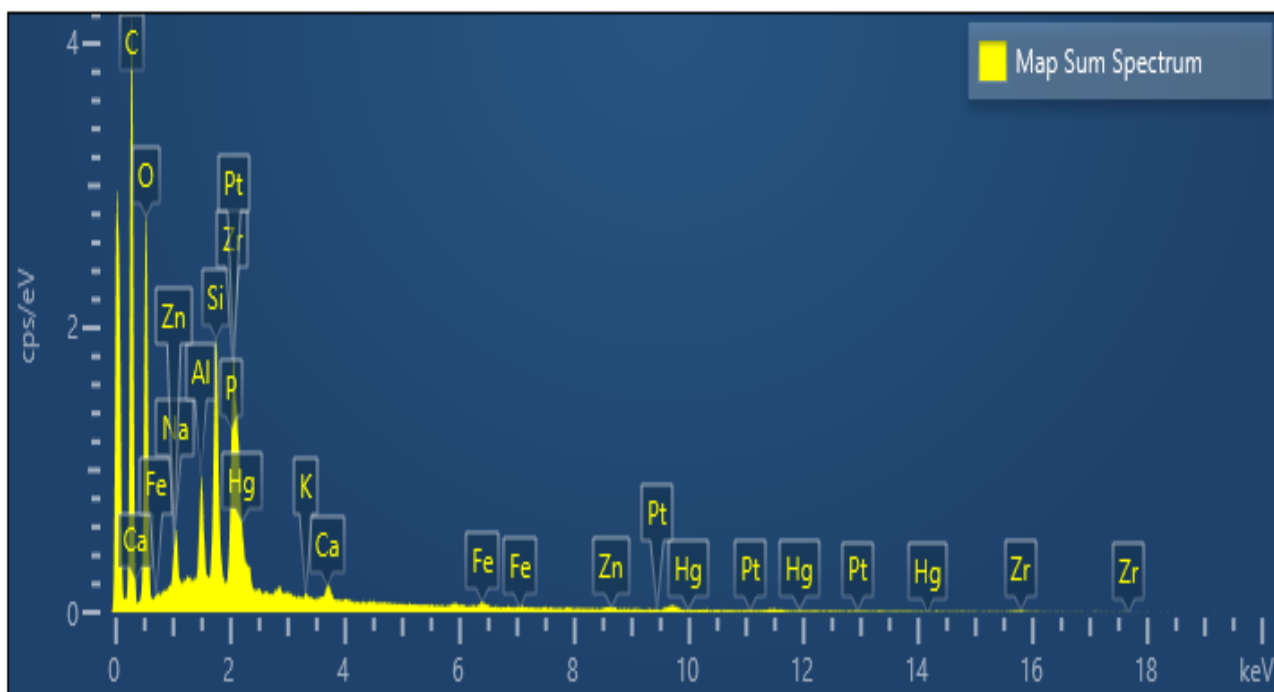


Fig.5.12: Map sum spectrum of rGO)

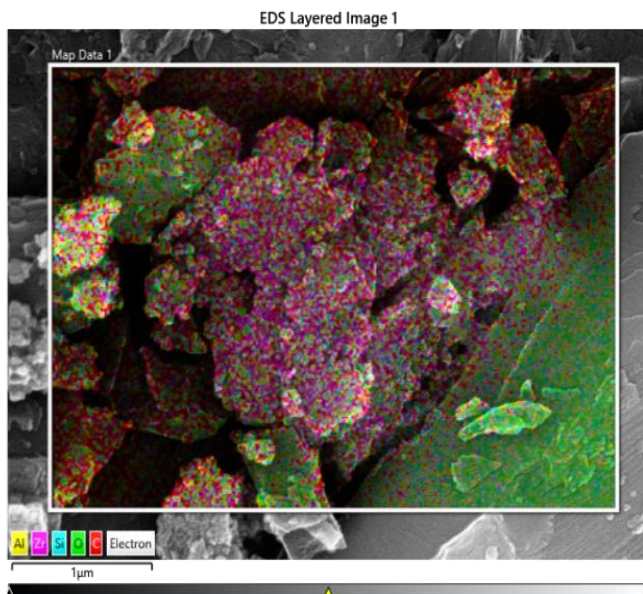


Fig.5.13: EDAX Elemental map

Table 5.1 Elemental Composition of rGO

ELEMENT	WEIGHT%
C	58.54
O	29.59
Na	0.91
K	0.52
Fe	0.24
Impurities	10.1
TOTAL	100

The analysis of the nanocomposite's Energy Dispersive X-ray Spectroscopy (EDS) data provides crucial information about its elemental composition

1. Percentage of Carbon (C) and Oxygen (O):

- Carbon (C): 58.54%
- Oxygen (O): 29.59%

2. Interpretation:

- The presence of both carbon and oxygen in the nanocomposite is consistent with the composition of reduced graphene oxide (rGO).
- Reduced graphene oxide typically contains carbon atoms bonded to oxygen functional groups, such as hydroxyl, epoxy, and carboxyl groups.
- The high percentage of carbon suggests a significant presence of graphene-based material, which aligns with the expected composition of rGO.

- The oxygen content is also substantial, reflecting the oxygen functional groups present in rGO.
- The C/O ratio obtained from the EDS data is consistent with reduced graphene oxide (rGO), further supporting the formation of the rGO nanocomposite with its characteristic elemental composition.
- The composition data obtained from EDS contributes to a comprehensive understanding of the nanocomposite's properties, which is essential for optimizing its performance in various applications, including skincare.

Zn Zr Reduced Graphene Oxide:

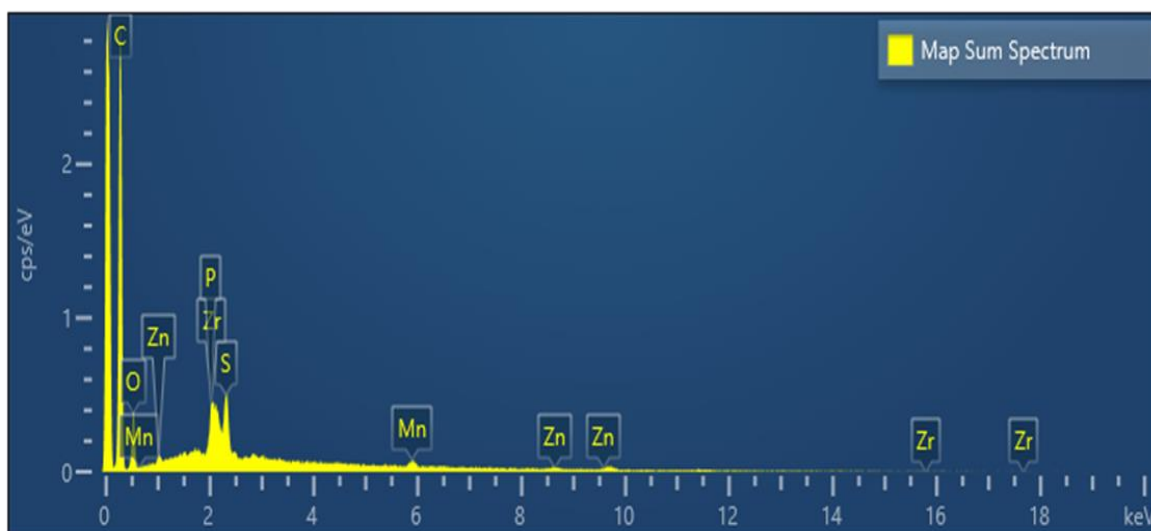


Fig.5.14: Map sum spectrum graph of Zr ZnO/doped rGO

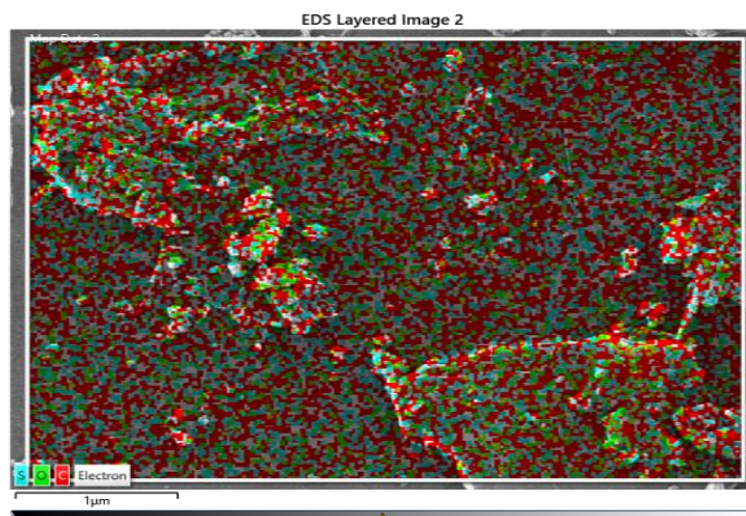


Fig.5.15: EDAX Elemental map

Table 5.2 Elemental Composition of Zr ZnO/rGO

ELEMENT	WEIGHT%
C	79.66
O	14.54
Mn	0.83
Zr	2.56
Zn	0.96
Impurities	1.45
TOTAL	100

The EDAX analysis of the rGO-Zr-doped ZnO nanocomposite offers valuable insights into

its elemental composition:

1. Detected Elements:

- The EDAX spectrum detected the presence of Zr (zirconium), Zn (zinc), C (carbon), and O (oxygen) in the nanocomposite.

2. Percentage of Elements:

- Zr: 2.56%
- Zn: 0.96%
- C: 79.66%
- O: 14.54%

3. Interpretation:

- The presence of Zr and Zn in the EDAX spectrum confirms the successful incorporation of Zirconium-doped Zinc Oxide into the nanocomposite. This validates the formation of the rGO-Zr-doped ZnO nanocomposite.
- The high percentage of carbon (C) in the nanocomposite (79.66%) suggests a significant presence of graphene or reduced graphene oxide (rGO). This aligns with the expected composition, as graphene-based materials are commonly used in nanocomposite formulations.
- The presence of oxygen (O) is also expected, as it is often associated with the functional groups present on graphene or RGO. These oxygen functional groups are introduced during the reduction process of graphene oxide to form RGO.
- Understanding the elemental composition is essential for tailoring the properties and performance of the nanocomposite for specific applications.
- The presence of Zr and Zn suggests potential applications in various fields such as photocatalysis, sensing, or antimicrobial applications, in addition to skincare.

5.16 X RAY DIFFRACTION

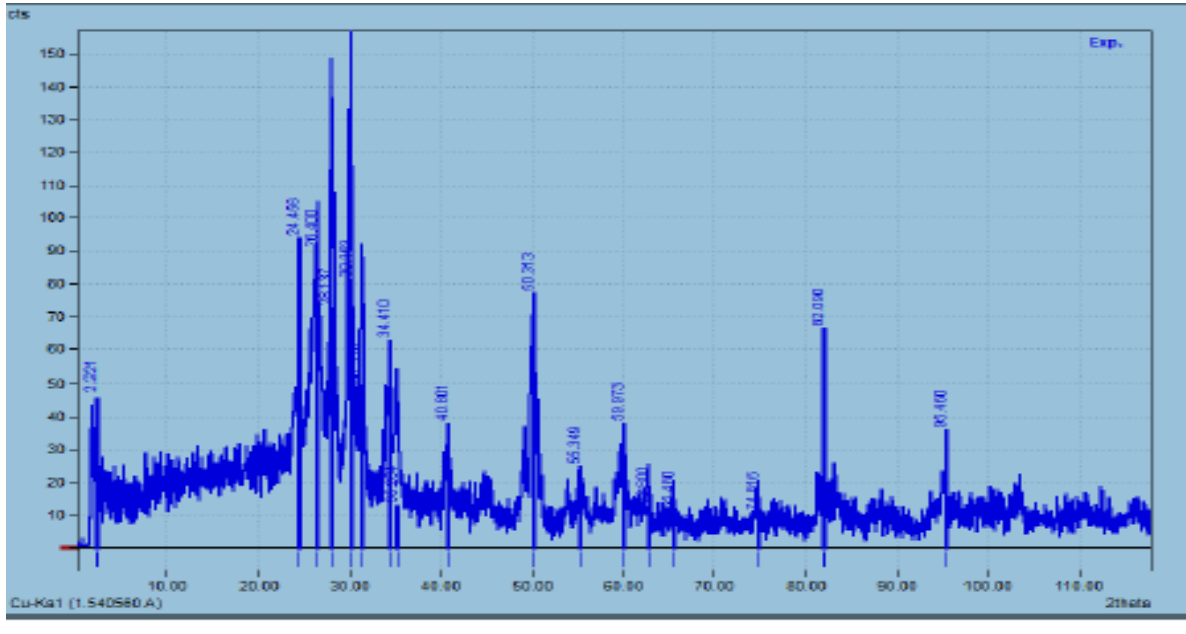


Fig. 5.16: XRD graph of Zr ZnO/rGO

1. Distinct Diffraction Peaks:

- The diffractogram displays distinct diffraction peaks at specific angles (2θ), indicating the presence of crystalline phases within the nanocomposite.

2. Cubic Crystal Structure:

- The observed diffraction peaks correspond to a cubic crystal structure, suggesting the crystalline nature of the nanocomposite.

3. Peak Intensity:

- The overall peak intensity in the XRD spectra is reported to be 820 counts, indicating high crystallinity. Sharp peaks further support this observation.

4. Highest Peak and D-Spacing:

- The highest peak is observed at 2θ of 30.17° , corresponding to a d-spacing of $2.95 \text{ \AA}$. This information provides insights into the interatomic spacing within the crystal lattice.

5. Nano Range Crystal Sizes:

- The XRD results confirm the synthesis of the ZrO₂-ZnO/rGO nanocomposite, with crystal sizes in the nano range, indicating particles less than 100 nm in size.

6. Implications:

- The XRD analysis confirms the crystalline structure of the rGO-ZrO₂-ZnO nanocomposite and identifies its cubic crystal structure.
- The sharp peaks and high peak intensity suggest a high degree of crystallinity, indicating well-defined crystallographic planes within the nanocomposite.
- The observation of crystal sizes in the nano range is advantageous for various applications in nanotechnology and materials science, including skincare, where smaller particle sizes can enhance properties such as absorption and efficacy.

5.17 FTIR SPECTROSCOPY

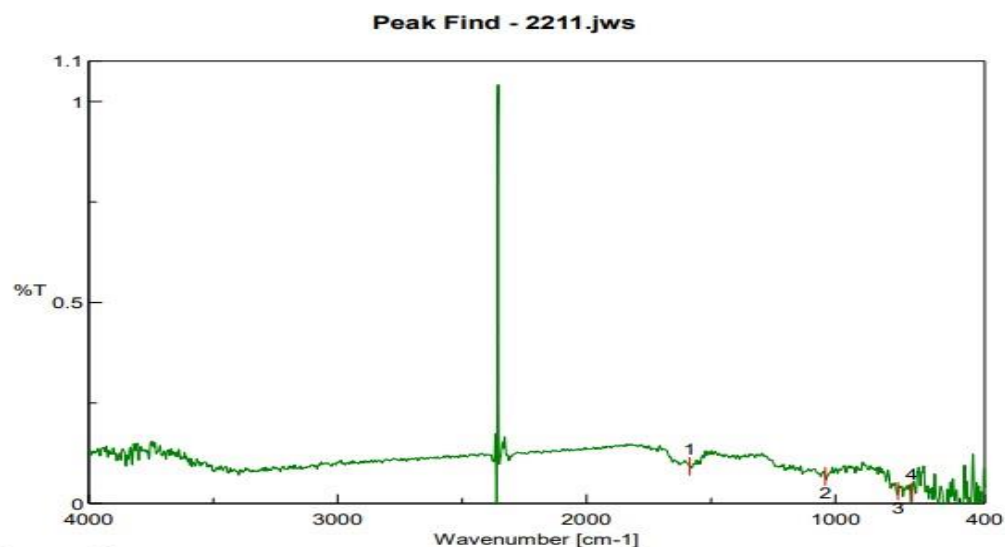


Fig. 5.17: FTIR spectroscopy of Zr ZnO/rGO

- Reduced graphene oxide (RGO) FTIR (Fourier Transform Infrared) spectroscopy investigation offers important information about the chemical and structural makeup

of RGO. Characteristic peaks are seen in the resultant spectrum, which represents the distinct bonds found inside the RGO lattice.

- Significant peaks are found in the 1600–1700 cm^{-1} region, which corresponds to the C=C stretching vibrations and indicates the presence of sp^2 hybridized carbon atoms that are intrinsic to graphene structures.
- Furthermore, the spectra might show peaks related to C-O and C-H bonds, which are generally located in the 1000–1300 cm^{-1} range and are suggestive of functional groups that still include oxygen from the original precursor of graphene oxide.
- Crucially, the lack or decrease in strength of peaks linked to oxygen functions indicates that graphene oxide has been successfully reduced to RGO. hydroxyl (OH) and epoxy (C-O-C) groups, in contrast to graphene oxide's FTIR spectrum.
- The restoration of graphene's sp^2 hybridization network, which is essential to its mechanical and electrical characteristics, is confirmed by this reduction.
- Moreover, any extra peaks or spectrum changes can point to possible RGO functionalization or doping, providing information about its specific chemical characteristics.
- All things considered, FTIR analysis is a vital method for describing the structural changes and chemical alterations that graphene experiences during the reduction process, which makes it easier for it to be used in a variety of technological applications with improved characteristics.

5.5 Barrett-Joyner-Halenda (BJH) Analysis

1. Surface Area (SA):

- Surface area refers to the total area of the material's surface per unit mass.
- A higher surface area indicates a greater number of active sites available for adsorption or reaction.
- In skincare, materials with higher surface areas are desirable for their potential to interact with skin or other ingredients, enhancing efficacy.

2. Pore Volume (PV):

- Pore volume represents the total volume of pores within the material per unit mass.
- It reflects the material's capacity to hold fluids or molecules within its pore structure.
- Skincare products may benefit from materials with appropriate pore volumes, as they can aid in the retention and release of active ingredients.

3. Pore Diameter Dv(d):

- Pore diameter refers to the average diameter of pores within the material.
- It provides insight into the size distribution of pores, with the specified value indicating the average diameter.
- Understanding pore diameter is essential for selecting materials suitable for specific skincare applications. For example, smaller pore diameters may be preferred for controlling the release of active ingredients, while larger pore diameters may facilitate better penetration into the skin.

Table 5.3 BJH desorption summary

Surface Area	45.760 m ² /g
Pore Volume	0.066 cc/g
Pore Diameter Dv(d)	2.488 nm

5.6 Brunauer-Emmett-Teller (BET) surface area analysis

1. Slope:

- The slope of the BET plot is related to the monolayer capacity of the adsorbent.
- It reflects the adsorption behavior of the material at low relative pressures, where monolayer adsorption predominates.
- The slope is used in the calculation of the specific surface area.

2. Intercept:

- The intercept of the BET plot is related to the volume of the adsorbate gas.
- It reflects the volume of gas required to form a monolayer on the material's surface.
- The intercept is also used in the calculation of the specific surface area.

3. Correlation Coefficient (r):

- The correlation coefficient indicates how well the experimental data fits the BET model.
- A value close to 1 suggests a good fit between the experimental data and the BET model.
- It helps assess the reliability of the specific surface area calculation.

4. Constant:

- This constant is used in the BET equation and is determined experimentally.
- It accounts for factors such as the properties of the adsorbate gas and the experimental conditions.

5. Surface Area:

- Surface area represents the specific surface area of the material, typically expressed in square meters per gram (m^2/g).
- It represents the total surface area available for adsorption per unit mass of the material.

- The specific surface area is calculated using the BET equation based on the slope, intercept, and constant obtained from the BET plot.

Table 5.4 BET summary

Slope	89.234 1/g
Intercept	1.564e+01 1/g
Correlation coefficient, r	0.998398
Constant	6.706
Surface Area	33.207 m ² /g

5.2 Evaluation of serum

5.2.1 Physical Appearance

The serum formulation is blackish grey in color, indicating its visual appearance. It has a viscous liquid consistency with a smooth homogeneous texture and a glossy appearance, suggesting a well-blended and visually appealing product.



Fig. 5.18: Physical Appearance Of rGO Face Serum

5.2.2 pH:

The pH of the formulation was measured at 4.24. Given that the skin has an acidic pH range of around 4.1-6.7, this pH falls within the suitable range for skincare products.



Fig. 5.19: pH of the serum

5.2.3 Globule Size Determination:

- A critical component of our analysis in our study of the reduced graphene oxide (rGO) serum was figuring out the dimensions of the globules were in the mixture. Using dynamic light scattering (DLS) or other comparable methods, we were able to carefully examine the globules and gain important information about their

distribution and average size.

- Our findings showed that the rGO serum's globule size varied within a particular range, usually staying within nanoscale dimensions. The accurate measurement of globule size is crucial since it affects the stability, texture, and effectiveness of the serum when applied topically.
- We improve our knowledge of the physical characteristics of the serum by measuring and describing the size distribution of these globules. This allows for necessary formulation modifications and additional optimization.
- These results add to the thorough evaluation of the rGO serum's applicability for skincare applications, guaranteeing that its performance and efficacy fulfill the required criteria

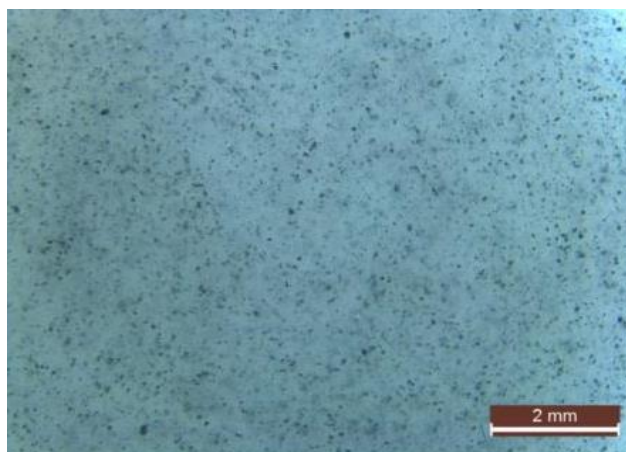


Fig. 5.20: Globule size of graphite serum in stereo microscope

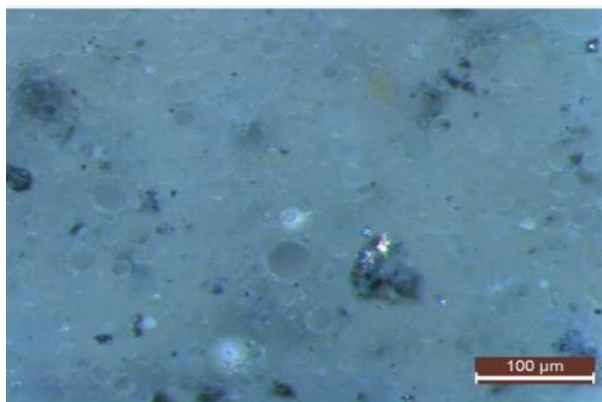


Fig. 5.21: Globule size of graphite serum in stereo microscope

5.2.4 Stability Studies:

- Our reduced graphene oxide (rGO) face serum is subject to extensive stability testing to guarantee its dependability and effectiveness. These tests are an essential part of determining if the serum can continue to function well and preserve its quality throughout time in a variety of environments.



Fig. 5.22: Stability test of rGOserum

- To assess the serum's physical characteristics throughout time, such as color, consistency, and odor, we first perform a stability test. This test makes sure the serum doesn't lose its aesthetic appeal or undergo any unfavorable modifications that can reduce its potency.
- In order to replicate actual storage circumstances, we also submit the serum to temperature changes in order to evaluate its resistance to external stresses. In order to make sure the serum keeps its active ingredients and is effective for the duration of its shelf life, we also examine the chemical makeup of the serum. To confirm the serum's ability to resist microbial contamination and protect against any health hazards, stability testing is also carried out.
- We can confidently produce a high-quality skincare product that satisfies the exacting standards of performance, security, and reliability by carefully evaluating

the stability of our rGO face serum.

5.2.5 MICROBIOLOGY TEST:

- To guarantee its safety and effectiveness, the reduced graphene oxide (rGO) face serum in our study was subjected to extensive microbiological testing. These tests' outcomes showed that there was no microbiological contamination in the serum, proving that it was suitable for use in skincare products
- Standardized methods were followed for performing microbiological assays, such as total aerobic microbial tally, mold, and yeast assays, and bacterial endotoxin tests.
- The lack of microbial growth in the serum samples shows how well our production method preserves the microbiological integrity of the product.
- These findings support the rGO face serum's potential as a dependable skincare product by giving stakeholders and customers confidence about the product's quality and safety.

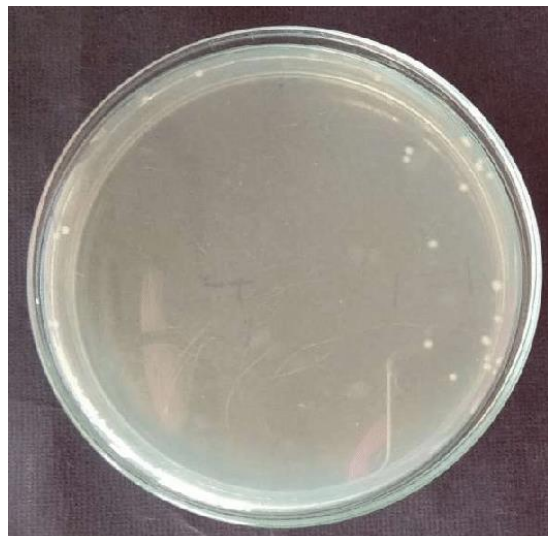


Fig. 5.23: Microbiology test of rGO serum

5.2.6 PATCH TEST:

The absence of observable skin reactions following a patch test with the serum suggests several important points regarding its safety and suitability for use:

1. **Non-allergenic Nature:** The lack of erythema (redness), edema (swelling), or vesicles (blisters) indicates that the tested individual did not manifest an allergic response to the serum. Allergic reactions typically involve the immune system's hypersensitive reaction to specific allergens, resulting in visible skin manifestations.

Therefore, the absence of such reactions suggests that the serum does not contain allergenic components or that the individual tested does not have an allergy to any of its ingredients.

2. **Non-irritating Formulation:** Similarly, the absence of any skin irritation, such as redness or swelling, suggests that the serum is unlikely to cause irritation or sensitivity in the tested individual. Skin irritation can occur due to various factors, including harsh chemicals, fragrances, or abrasive substances in skincare products. The lack of observable irritation indicates that the serum formulation is gentle and well-tolerated by the skin.

3. **Safety for Use:** The overall interpretation of the patch test results leans towards the serum being safe for use on the skin of the tested individual. This implies that the serum can be considered suitable for inclusion in their skincare routine without significant risk of adverse reactions. However, it's important to note that individual skin sensitivities can vary, and ongoing monitoring is advisable, especially if the serum is used over an extended period.

CHAPTER 6

SUMMARY AND CONCLUSION

6.1 SUMMARY

In this work, the synthesis, characterisation, and application of bimetallic reduced graphene oxide (rGO) and its formulation as a facial serum are thoroughly examined. By employing a bimetallic catalyst to reduce graphene oxide (GO), bimetallic rGO was created, a material with improved characteristics fit for a variety of uses. The structural, chemical, and surface properties of the bimetallic rGO were analyzed using characterization techniques like scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET) surface area analysis, and Barrett-Joyner-Halenda (BJH) pore size distribution analysis. The outcomes showed that bimetallic rGO with specific structural and surface properties had been successfully synthesized, making it appropriate for a wide range of uses. Additionally, the rGO preparation In order to maximize the benefits of its distinct features for better skincare, bimetallic rGO was incorporated into a face serum composition.

6.2 CONCLUSION:

The synthesis and characterisation of bimetallic reduced graphene oxide (rGO) present exciting prospects for a range of uses, including the development of skincare formulations. SEM, EDX, FTIR, XRD, BET, and BJH were some of the characterisation methods used. These methods gave important information about the surface, chemical, and structural characteristics of bimetallic rGO. These analyses made it possible to incorporate bimetallic rGO with customized features into cosmetics formulations by confirming its successful production. The creation and analysis of rGO face serum revealed its potential to improve skincare products by utilizing the special qualities of bimetallic rGO. It is necessary to conduct additional study and development in this area going forward. Investigate bimetallic rGO's complete potential in skincare and other uses, as this will ultimately help both consumers and businesses

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**“SYNTHESIS AND APPLICATION OF ZIRCONIUM DOPED ZINC OXIDE
REDUCED GRAPHENE OXIDE IN SKINCARE”**

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology degree in Chemical Engineering

By:

SNEHA S (40190026)
G DIVYANJALI (40190008)



**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING**

SATHYABAMA

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of SNEHA S(40190026) and G DIVYANJALI (40190008) who carried out the project entitled "SYNTHESIS AND APPLICATION OF ZIRCONIUM DOPED ZINC OXIDE REDUCED GRAPHENE OXIDE IN SKIN CARE" under my supervision from _____ to _____

Internal Guide

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Submitted for Viva-voce Examination held on _____

Internal Examiner

External Examiner

DECLARATION

We SNEHA S (40190026) and G Divyanjali (40190008) hereby declare that the project Report entitled "SYNTHESIS AND APPLICATION OF ZIRCONIUM DOPED ZINC OXIDE REDUCED GRAPHENE OXIDE IN SKINCARE" done by us under the guidance of Dr. SATHISH S M.E.,PH.D., at SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY is submitted in partial fulfillment of the requirements for the award of BACHELOR OF TECHNOLOGY DEGREE IN CHEMICAL ENGINEERING.

DATE:

PLACE:

SIGNATURE OF THE CANDIDATES

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ABSTRACT

This study investigates the biocompatibility of bimetallic reduced graphene oxide (rGO)-based functional nanomaterials. rGO doped with zinc oxide and zirconium dioxide (rGO-ZnO-ZrO₂). The purpose of this effort is to develop a serum that uses the emerging nanomaterial as an active element in skincare. This article describes a method for producing bimetallic reduced graphene oxide using modified Hummers processes. The reduction of GO to rGO was accomplished with the help of L-ascorbic acid. The characteristics of both rGO and Zn Zr/rGO were thoroughly studied utilizing X-ray diffraction (XRD), scanning electron microscopy (FE-SEM), energy dispersive X-ray analysis (EDX), Fourier-transform infrared spectroscopy (FTIR), Brunauer-Emmett-Teller (BET) surface area analysis, and Barrett-Joyner-Halenda (BJH) pore size distribution analysis. And evaluating the graphite serum by its chemical and physical properties. The primary purpose of this effort is to explore the potential of bimetallic rGO-based nanomaterials as active elements in skincare formulations, and to pave the way for the development of innovative skincare products that leverage the unique properties. This research contributes to advancing the knowledge and application of nanotechnology in skincare, offering new opportunities for the formulation of effective and sustainable skincare solutions.

KEYWORDS: Reduced Graphene Oxide, Zirconium doped Zinc Oxide Reduced Graphene Oxide, rGO serum

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LIST OF ABBREVIATIONS

FE-SEM	Scanning Electron Microscope
FTIR	²⁵ Fourier Transform Infrared Spectroscopy
EDAX	Energy Dispersive X-Ray Analysis
XRD	X- Ray Diffraction
BJH	Barrett-Joyner-HalendaPore Size Distribution Analysis
BET	Brunauer-Emmett-Teller Surface Analysis
GO	⁵¹ Graphene Oxide
rGO	Reduced Graphene Oxide
Zr ZnO/rGO	Zirconium doped Zinc Oxide Reduced Graphene Oxide

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CHAPTER 1

INTRODUCTION

The investigation of novel materials for biomedical applications has attracted a lot of attention lately, especially in the areas of therapeutic interventions, drug delivery, and diagnostics. Reduced graphene oxide is one of these compounds that stands out as a versatile material with remarkable adsorption capabilities. Charcoal, which has long been used in air filtration, water purification, and poison countermeasures, is now being used in the biomedical industry because of its capacity to adsorb infections, poisons, and other organic molecules.

Given this, creating rGO serum seems like a fresh and exciting approach that combines the benefits of reduced graphene oxide with the adaptability of serum-based therapies. Reduced graphene oxide particles are either suspended in or incorporated into rGO serum, a novel formulation. Reduced graphene oxide particles are either suspended in or incorporated into a serum matrix in rGO serum, a novel formulation that provides a platform for specific biological functions and customized adsorption capacities. The purpose of this work is to investigate the characterisation methods, production processes, and possible uses of rGO serum in biomedicine. We want to provide a thorough understanding of this developing material by exploring the nuances of its production process, clarifying its physicochemical features, and evaluating its performance. A number of factors must be taken into account when creating rGO serum, such as choosing suitable sources of charcoal, perfecting integration or suspension methods, and adjusting serum composition to fulfill the required functions.

Additionally, the characterisation of rGO serum requires the use of a number of analytical instruments, including surface area analysis, Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM), to clarify its adsorption capabilities, surface characteristics, and structural characteristic.

This study examines the various uses of rGO serum in biomedicine in addition to its manufacture and characterization. rGO serum has the potential to be used as a medication delivery vehicle and is useful in detoxification therapy and diagnostic assays. It can be used to address a wide range of biomedical issues. Its variable adsorption capabilities and compatibility with biological systems further make it a desirable candidate for targeted therapeutic interventions.

All things considered, the production of rGO serum is an intriguing example of the blending of material science and biology, providing a flexible platform with a broad range of uses. By expanding our knowledge of its production processes, characteristics, and biomedical features, we open doors for the creation of novel approaches in biomedical research, diagnostics, and medication delivery. Our goal in publishing this study is to encourage more investigation and research in this emerging subject, which will ultimately lead to improvements in biotechnology healthcare. In order to create reduced graphene oxide (rGO), graphene oxide (GO) from natural graphite is usually prepared using oxidation techniques like the Hummers' method. To do this, strong oxidizing chemicals are applied to graphite in order to introduce functional groups that contain oxygen. The reduction of GO to rGO is then accomplished by dispersing GO in an appropriate solvent and adding a reducing agent, such as ascorbic acid or hydrazine. Applying heat or using ultrasonication speeds up the reduction process, which removes oxygen functions and sp² hybridization recovery. After that, all remaining compounds and impurities are removed from the resultant rGO through purification.

Zn-Zr doping of rGO entails the surface implantation of zirconium-doped zinc oxide (ZnO) nanoparticles. This is accomplished by dissolving zinc and zirconium salts in a solvent to create a precursor solution. The required doping concentration determines the molar ratio of zinc to zirconium. After mixing the ZnO precursor solution with the rGO suspension, the ZnO nanoparticles are encouraged to develop on the rGO surface using hydrothermal or solvothermal treatment.

Through the introduction of zirconium doping, this technique improves the electrical, optical, and structural features of rGO for a range of applications.

The synthesized rGO is characterized by evaluating its structural, morphological, and chemical properties using a range of analytical methods. X-ray diffraction (XRD) for crystalline structure analysis, Raman spectroscopy for assessing structural defects and reduction in degree, scanning electron microscopy (SEM) or transmission electron microscopy (TEM) for morphological analysis, and Fourier-transform infrared spectroscopy (FTIR) for identifying functional groups are examples of common characterization techniques.

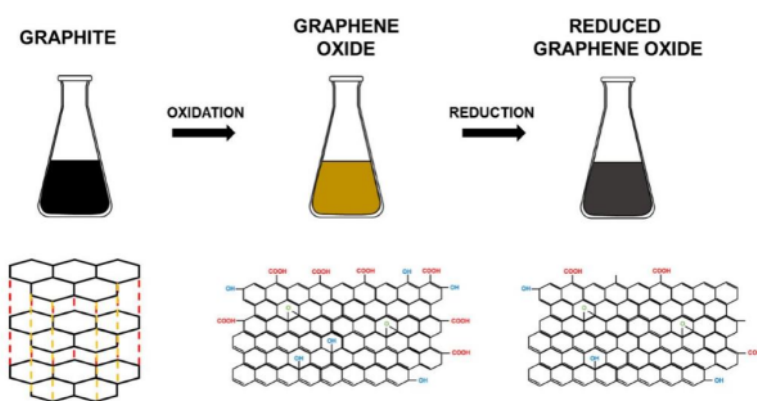


Fig 1.1 General representation of reducing GO to rGO

These characterisation methods offer important information about the composition and quality of the produced ZrZnO/rGO. In order to create the face serum using an rGO basis, the right ingredients, including water, humectants, emollients, and active compounds, must be chosen to create a proper formulation. The previously produced rGO/ZnO nanocomposite is distributed throughout the serum basis to provide stability and uniform distribution. The desired skincare features determine the incorporation of additional active components like antioxidants, moisturizers, peptides, or botanical extracts. When necessary, the formulation's pH and viscosity are changed to maximize product performance and user satisfaction.

In order to assess the face serum's effectiveness, stability, and safety, a thorough analysis is performed at the end. In vitro and/or in vivo models are used for skin compatibility and irritant testing, while stability studies are carried out under various settings to evaluate shelf life. Physical characteristics including color, smell, and viscosity are tracked over time, and methods are used to measure the amount of active substances. Consumer trials collect input from human participants to evaluate the effectiveness and user experience, guaranteeing that cosmetic goods meet safety regulations and legal requirements. Reduced graphene oxide (rGO) is being synthesized for more than just material production; the goal is to raise awareness about the potential of 2D nanomaterials and skincare uses.

rGO synthesis provides a versatile method that supports this goal. First off, the procedure has inexpensive instrumentation, making it accessible to both practitioners and researchers. Because of its cost, the field of nanoparticles can be extensively explored and experimented with, leading to innovation and discovery. Large product outputs from the procedure also guarantee a steady supply for skincare formulation testing.

Moreover, rGO synthesis's eco-friendliness emphasizes its importance in today's sustainability initiatives. Green chemistry principles are applied in rGO synthesis to minimize environmental impact and to be in line with the increasing worldwide emphasis on sustainable practices. Furthermore, rGO can be produced using the synthesis approach in a variety of morphological forms and sizes, providing flexibility for use in skincare applications. Because of its versatility, researchers can customize rGO characteristics to meet certain skincare requirements while maximizing effectiveness and performance.

The fact that rGO synthesis doesn't require pricy catalysts or hazardous surfactants is another noteworthy benefit. This lowers production costs while guaranteeing the creation of high-quality products devoid of potentially dangerous impurities.

GO synthesis makes it easier to produce skincare formulas that satisfy demanding quality requirements and customer expectations by putting safety and product integrity first.

Using zinc-zirconium (Zn-Zr) doping to synthesize bimetallic reduced graphene oxide (rGO) offers a viable way to improve the characteristics of graphene-based materials used in skincare products. Graphene oxide (GO) is reduced to reduced graphene oxide (rGO) by means of ascorbic acid, which is selected due to its economical and eco-friendly characteristics. As an effective reducing agent, ascorbic acid helps to remove oxygen-containing functional groups from graphene oxide (GO) with the least amount of negative environmental effects.

This environmentally friendly method fits nicely with the growing focus on sustainability in the synthesis-materials. There are several possible advantages to adding rGO to skincare formulas. Because of its distinctive structural characteristics, such as strong electrical conductivity and surface area, rGO shows great promise for improving the effectiveness of skincare solutions. The resulting bimetallic rGO composite can further improve its antibacterial activity, UV protection, and antioxidant qualities by doping it with Zn-Zr. These improvements are especially beneficial for skincare, as it's critical to preserve skin health and shield it from outside-stresses.

The effectiveness and efficacy of skincare products can be enhanced by including rGO. For instance, increased skin hydration, elasticity, and attractiveness may result from rGO's capacity to improve skin barrier function and stimulate collagen formation. Its antibacterial qualities can also aid in the fight against bacteria and other pathogens that cause acne, promoting healthier, cleaner skin. Additionally, Zn-Zr dopants may offer other advantages including improved

Antioxidant activity and UV protection also support the health and protection of skin. All things considered, creating bimetallic rGO doped with Zn-Zr is a viable method of incorporating cutting-edge nanomaterials into skincare formulas. Researchers and product developers can take advantage of the special qualities

of reduced graphene oxide (rGO) to build cutting-edge skincare solutions that improve overall skin health, performance, and protection. This can be achieved by utilizing the economical and ecologically friendly reduction. The name "serum," which refers to a highly concentrated product that is frequently used in cosmetology, comes from its professional use. Serums are 10 times more organic than ordinary creams, which helps them address cosmetic issues quickly and effectively. The need for cosmetic items has surged due to the rising worldwide cost of living, hence situating the cosmetics business.

As more people strive to stay young and appealing, cosmetics have become more and more valuable. Whether in the form of a gel, light lotion, or moisturizer, a skin serum has the capacity to deeply permeate the skin and supply the skin with active chemicals. It can reduce pores, increase moisture content, and improve the firmness and texture of the skin. Antioxidants, chemicals derived from cells, and substances resembling skin are essential for preserving skin health in all forms. Richer moisturizing creams are advised for dry to extremely dry skin, while serums and light lotions work best for normal to dry skin. Gel formulations and liquids are ideal for oily or combo skin. The essential components for skin health are the same in all products, regardless of differences in skin texture. The largest and most defensive organ in the body is the skin, can become dry as a result of a number of things, including allergies, debris, UV rays, and leftover makeup.

Because serum has small molecules, it may be applied directly to specific skincare concerns like uneven skin tone or age indications. Applying serum after cleansing but before moisturizing allows for this.

Cosmeceuticals are skincare products that combine cosmetic and medical characteristics. They are a blend of cosmetics and pharmaceuticals. As defined by the Health Sciences Authorities' Guide to the Control of Cosmetic Products (Revised 2014), a cosmetic product is anything that is meant to be applied to the skin, hair, nails, lips, eyes, teeth, and external genitalia in order to clean, perfume, change appearance, control odor, or provide protection. Usually, these products fall into one of five primary categories. Enhancing the skin's barrier function, an

emulsion-based face serum serves as a moisturizer and a delivery system for powerful skincare products. Emulsions combine two phases that don't seem to go together, like water and oil, by stabilizing the mixture with emulsifiers. This makes it possible to produce a stable product that successfully blends the advantages of the two stages.

An emulsion-based serum's primary benefit is its capacity to introduce active ingredients deeply into the skin's tissues. This is important since the skin's natural barrier makes it difficult for elements in cosmetics to penetrate deeply. Through the use of water and oil in an emulsion, the serum works to increase the probability that these potent active ingredients will reach the dermis and begin to work. Most successfully. Additionally, the emulsions' natural moisturizing qualities reinforce the skin's barrier function, promoting the general resilience and health of the skin. To ensure the serum's efficacy, safety, and quality in skincare compositions, each test that is performed on it is vital. By detecting and measuring any microbiological contamination, this analysis guarantees that the serum is free of dangerous pathogens that could infect skin or produce unfavorable reactions when applied topically.

Studies on the serum's stability are carried out to evaluate its capacity to retain quality overtime the physical properties and assessment of the rGO serum concentrate on identifying any alterations or distinctiv First and foremost, the spreadability test is important since it assesses how readily the serum can be administered and dispersed throughout the surface of the skin. Uniform coverage and absorption are ensured by a serum with ideal spreadability, which improves user experience and maximizes the administration of active ingredients, determining the reduced graphene oxide (rGO) serum's microbiological safety requires a microbial analyse features related to the presence of reduced graphene oxide. This assessment offers important insights into how rGO is incorporated into the serum matrix, guaranteeing its efficacy and suitability for use in skincare products. stability and compatibility. It affects the effectiveness of the active components in the serum as well as the skin barrier function and irritation

potential. To maximize the serum's benefits and reduce the chance of skin irritation, make sure its pH is within the physiological range of the skin.

Lastly, the viscosity measurement contributes to our understanding of the consistency and texture of the serum, which influences its spreadability, application ease, and skin absorption. Formulators can obtain the required product attributes and ensure optimal user experience and efficacy by assessing viscosity and adjusting the formulation accordingly.

All things considered, the results of these serum tests collectively offer thorough insights into the product's stability, safety, and effectiveness, guaranteeing the creation of superior skincare products that satisfy both legal and consumer requirements. A noteworthy advancement in skincare technology is the development of arGO (reduced graphene oxide) serum. Through the utilization of rGO's extraordinary characteristics, such as its large surface area, high conductivity, and biocompatibility, we have the potential to completely transform skincare formulas. With these qualities at our disposal, we want to develop innovative skincare products that successfully address a wide range of cosmetic issues.

Our mission is to fully realize the potential of rGO-based serums with a rigorous testing protocol and meticulous experimentation process. Our attempts to bring in a new era of skincare innovation are based on this commitment to scientific study and quality assurance. Our ultimate goal is to launch next-generation skincare products that not only offer unmatched effectiveness while giving sustainability of the environment and safety equal priority. By utilizing rGO, we hope to raise the bar for the skincare sector and enable people all around the world to have healthier, more beautiful skin.

The groundwork for our novel skincare serum is laid during the first part of our formulation process, where we carefully prepare the oil and water phases, to aid in emulsification. We provide the foundation for future investigations and

understandings of the serum's functionality and effectiveness by thoroughly evaluating the serum's globule size and physical characteristics

When graphene oxide is heated or chemically treated, ⁴¹reduced graphene oxide (rGO) is produced. This is a noteworthy development in the field of material science. This process uses reducing agents such as sodium borohydride, l-ascorbic acid, hydrazine, and hydrazine hydrate to occur between graphite layers and oxidized graphene oxide. Notably, recent studies have investigated the application of plant extracts for rGO synthesis, taking advantage of their accessibility and friendliness.

These extracts help to produce nanoparticles by acting as capping and reducing agents. Furthermore, rGO's functional groups allow for a variety of chemical interactions with biomolecules, including enzymes, proteins, peptides, and DNA. Biomolecules are being used more often in rGO synthesis due to their low labor costs, biocompatibility, and potential uses in a variety of industries, including medicine. Additionally, rGO's low toxicity increases its potential. Its high surface area enhances its effectiveness when used therapeutically.

Through a variety of methods, composites of rGO with metals and metal oxides have demonstrated great potential in improving supercapacitor performance. In order to create rGO nanocomposites embellished with Ag and MnO₂ nanoparticles, for example, Ma et al. presented a two-step synthesis process that produced uniformly distributed structures favorable to improved performance in energy storage applications. This process demonstrated the adaptability and potential of rGO in composite materials by first developing MnO₂ nanostructures on GO sheets in situ, then reducing GO and Ag⁺ to create Ag/MnO₂/rGO composites.

In many scientific and industrial applications, reduced graphene oxide (rGO), a crucial substance that is produced by reducing graphene oxide through chemical or thermal methods, fills a large role. The reduction process usually occurs between graphite layers and oxidized graphene oxide, using a variety of agents

including sodium borohydride, l-ascorbic acid, hydrazine, and hydrazine hydrate. But because plant extracts are readily available and environmentally benign, efforts in this area have recently shifted to using them. The addition of biomolecules not only speeds up the reduction process but also gives the final rGO biocompatible characteristics that make it suitable for a wide range of biomedical uses. Because of its low labor intensity and cost-effectiveness, this combination of reduced graphene oxide and biological entities has attracted interest as a promising. Biotechnology and materials science avenue. Furthermore, rGO's low toxicity by nature enhances its therapeutic potential and makes it easier to use in biomedical domains.

In addition to being biocompatible, rGO's large surface area has led to its incorporation into composite materials, increasing its usefulness in a variety of applications, including catalysis and energy storage. Recent studies, for example, have concentrated on creating rGO-based nanocomposites using complex synthesis techniques, producing materials with improved qualities for supercapacitor applications. Silver (Ag) and manganese dioxide (MnO₂) nanoparticles are evenly distributed onto rGO sheets in a two-step procedure that synthesizes a rGO nanocomposite.

This example illustrates the possibility for customized fabrication techniques to fine-tune the attributes of rGO composites. This expanding field of study highlights the adaptability and expanding potential of rGO in tackling current issues in a variety of multidisciplinary fields, such as energy storage friendliness. Regarding and biomedicine, and opening the door for creative technical developments. In recognition of its special qualities and adaptability, reduced graphene oxide (rGO) has attracted a lot of attention lately for use in a variety of applications. The synthesis, characterisation, and use of rGO in face serum compositions are the main topics of this research. rGO, which is produced by thermally or chemically treating graphene oxide (GO), is a desirable option for biomedical applications because to its exceptional electrical conductivity, high surface area, and biocompatibility.

In order to create rGO, oxygen-containing functional groups in GO must be reduced. This restores the sp² carbon network and creates a highly conductive material. For this reason, reducing agents including sodium borohydride, l-ascorbic acid, hydrazine, and hydrazine hydrate are frequently utilized. Numerous techniques, including chemical For the synthesis of rGO, reduction, thermal reduction, and green synthesis utilizing plant extracts have all been investigated; each has special benefits in terms of scalability, affordability, and environmental friendliness.

Regarding face serum compositions, rGO has a number of possible advantages, such as improving the absorption of active compounds into the skin, encouraging the production of collagen, and possessing antioxidant qualities.

Furthermore, the large surface area of rGO offers plenty of room for the loading and long-term release of bioactive substances, enhancing the stability and effectiveness of face serums.

we offer a thorough investigation into the environmentally friendly synthesis of rGO employing plant extracts as reducing agents. Using cutting-edge analytical methods, we describe the synthesized rGO and assess its potential use in face serum compositions. Additionally, we look into The findings of in vitro and in vivo studies on the impact of rGO incorporation on the physicochemical characteristics and functionality of face serums. The overall goal of this research is to use the special qualities of rGO to help produce novel and efficient skincare solutions.

In the realm of skincare, innovations continually emerge to address various skin concerns and deliver targeted benefits. Among these innovations, charcoal serum stands out as a potent and versatile solution for achieving healthier, clearer, and more radiant skin.



Fig 1.2 Skin with freckles and dark spots

Graphite serum represents a groundbreaking approach to skincare, harnessing the purifying and detoxifying properties of graphite to deeply cleanse and rejuvenate the skin. As a skincare product applied after cleansing and before moisturizing, graphite serum is formulated to deliver concentrated amounts of active ingredients to the skin, providing rapid and effective results.

Key Features of graphite Serum:

1. **Purifying and Detoxifying:** graphite acts as a magnet for impurities, toxins, and excess oil, drawing them out from deep within the pores and leaving the skin thoroughly cleansed and refreshed.
2. **Deep Hydration:** Despite its powerful detoxifying properties, graphite serum also provides deep hydration to the skin, helping to maintain moisture balance and prevent dryness.
3. **Skin Brightening:** graphite serum may contain brightening agents that help to ²⁹even out skin tone, reduce the appearance of dark spots and hyperpigmentation, and promote a more radiant complexion.

4. Anti-Acne Properties: graphite serum can be beneficial for those prone to acne or blemishes, as it helps to unclog pores, reduce inflammation, and prevent future breakouts.
5. Anti-Aging Benefits: Some graphite serums may also contain anti-aging ingredients such as antioxidants and peptides, which help to combat the signs of aging, including fine lines, wrinkles, and loss of elasticity.

In conclusion, the synthesis of reduced graphene oxide is a driving force behind the growing knowledge and comprehension of two-dimensional nanomaterials and their possible uses in skincare.

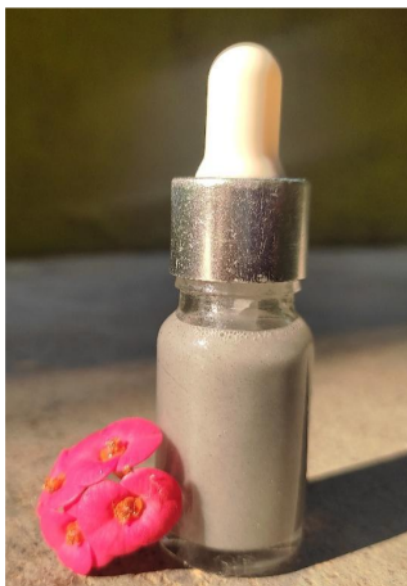


Fig 1.3 rGO face serum

Because of its inexpensive instrumentation, rGO synthesis opens the door for cutting-edge skincare products that are sustainable and effective thanks to its high product yield, eco-friendly manufacturing technique, and capacity to create high-quality, customization nanomaterials. The unique qualities and formulation of the RGO face

serum offer a promising range of skincare advantages. This serum is an adaptable way to help with different skin issues and support general skin health. RGO's anti-aging qualities are one of its main purposes; being high in antioxidants, it fights free radicals and lessens ¹³the appearance of wrinkles, fine lines, and age spots¹³. Its superior moisture-trapping ability further adds to skin hydration, making it a great option for anyone with dry or dehydrated skin. Moreover, RGO has skin-brightening qualities that improve complexion radiance and even out skin tone. Because of its anti-inflammatory properties, it's very useful for relieving discomfort, calming inflamed skin, and lowering redness. Furthermore, RGO's antibacterial qualities make it useful in the fight against germs that cause acne, which makes it an important part of acne treatment plans. All things considered, adding RGO face serum to regular skincare regimens can result in healthier, more radiant skin by addressing a variety of issues like aging indications, dehydration, and imperfections.

CHAPTER 2

LITERATURE SURVEY

- **Kanchan Chande¹ , Sunidhi Dessai² , Santosh Bhutekar³ , Md Talat Choudhary⁴ , Kamlesh Choudhary⁵ , Shriram Bairagi⁶ , Smita Takarkhede** have published a scientific article ; This essay focuses on the traditional application of herbal medicine, specifically chrysanthemum flower, for medicinal and skincare purposes. This practice is common not only in India but also in other parts of the world. Given that chrysanthemum flowers are rich in vitamins, flavonoids, carotenoids, anthocyanins, and essential oils—all of which have multiple advantages for skin health—the flower's inclusion in skincare products makes sense. The discovery of chrysanthemum's antibacterial and anti-inflammatory qualities highlights the plant's potential for treating common skin conditions like acne and inflammation brought on by UV radiation & photo damage.

In addition, the creation of a face serum with glycerin, vitamin E, and chrysanthemum is a good skincare strategy. Concentrated formulations of face serums are well-known for enabling deeper penetration into the skin layers, optimizing the active ingredient delivery. Due to its antioxidant qualities, vitamin E is included, which increases the serum's ability to fight environmental damage and aging of the skin. Though traditional herbal medicines have been used for many years, it's important to remember that their safety and efficacy need to be confirmed by rigorous research and clinical trials. Additionally, a number of variables, such as the formulation, the concentration of active substances, and the type of skin, affect how effective skincare products are.

This research concludes by highlighting the potential of chrysanthemum flower in cosmetics formulations and emphasizing the value of investigating natural components for their possible medicinal uses. Additional study in this field may result in the creation of secure and efficient.

- Sanjana Patil¹ , Akshada Deshmukh² , Dr. S.V Patil³ Prajakta Patil⁴ , Riya Patil⁵ , Pratiksha Patil⁶ , Shivtej Patil⁷ , Sayali Hogade** have published the paper The creation of a serum with a polyherbal extract made of coconut oil, almond oil, glycerin, and aloe vera gel is covered in the paper. The purpose of adding these chemicals to the serum recipe is to take advantage of each one's unique skincare benefits. The ability of aloe vera extract, which is well-known for increasing collagen, to reduce aging symptoms including wrinkles and fine lines is underlined. Liquorice, which contains glycyrrhizin, is also known for its antioxidant qualities, ¹³ which can help shield the skin from UV rays and free radical damage, thereby delaying the aging process. The serum's high concentration of active chemicals, deep penetration into the skin's layers, rapid absorption, and non-greasy finish are all said to contribute to its efficacy. The application The antioxidant benefits of coconut oil and its ability to prevent premature skin aging are also highlighted. All things considered, the study offers insightful information about how to create a serum that is enhanced with organic components that are well-known for their positive effects on aging and skin health. Nevertheless, additional investigation and clinical trials could be required to confirm the effectiveness and safety of the designed serum for use in skincare applications.
- Shan Sasidharan^{1*}, Pyarry Joseph ², Junise** have published a paper The formulation of a fairness serum using polyherbal extracts is presented in the study, with special attention to the serum's high concentration of active ingredients and quick skin absorption. Because of its noticeable effects, the serum promises to give not only rapid cosmetic results but also psychological happiness after treatment. Known for their ability to enhance fairness, a blend of active natural components, including Glycyrrhiza glabra, Crocus sativus, rice bran, and olive oil, are included in the formulation. The serum's capacity to permeate the skin's color cells and epidermis, improving skin tone and resulting in a fairer complexion, is emphasized. To further guarantee the formulation's safety and effectiveness, extensive analyses, including physicochemical and biological assessments, were conducted. The lack of heavy metals, high spreadability, and non-sensitizing qualities of the substance made it appropriate for employ. All things considered, the study provides insightful information about the creation of a fairness serum using polyherbal extracts, which may be helpful for

skincare lovers looking to achieve noticeable and durable improvements in skin tone and texture. The efficacy and safety profile of the serum may need to be confirmed through additional research and clinical studies before it can be widely used in skincare applications.

- **Tejal Shirish Shejul^{1*} Prof. Kiran Kudale^{1*}** has published a paper The study discusses the rising demand for skincare treatments and products and stresses how crucial it is to uphold appropriate look and adhere to modern beauty standards. It emphasizes the value of adding serums to skincare regimens and points out how adaptable they are in terms of meeting the needs of many skin types and issues. In the literature study, the advantages of utilizing serums with particular components designed to meet the demands of each different skin type—like brightening and healing qualities—are highlighted. It also addresses the possibility of using concentrated vitamin and acid serums to treat skin aging issues with encouraging outcomes. All things considered, the research offers insightful information about the function of serums in skincare routines and the effectiveness of specialty formulations in reaching targeted skincare results. But more investigation and clinical trials are needed. May be required to confirm whether serums are beneficial in treating particular skin issues and delaying the aging process.
- ¹⁹ **Smriti Ojha, surabhi sinha, swadhapiya das chaudhuri, hina chadha** have published a paper The article tackles the prevalent issues of wrinkles on the face and skin aging due to UV exposure and photodamage, highlighting the deficiency of efficacious methods to counteract the aging process. By creating a highly concentrated face serum with glycerin, honeybee venom, and aloe vera, it presents a fresh technique. Aloevera is emphasized for its many uses in skincare products because of its quick absorption, capacity to reach deeper skin layers, and medicinal qualities, which include antibacterial, anti-inflammatory, and wound-healing capabilities. Moreover, the serum composition gains a novel dimension from the incorporation of honeybee venom, which is well-known for its purported capacity to enhance blood flow and collagen formation. The assessment of the serum's physicochemical properties is covered in further detail in the publication, including pH, globule size, uniformity, and stability study

findings showing no appreciable variations in globule size or visual appearance over time. In summary, the study offers a potentially fruitful path for innovative skincare by utilizing the combined benefits of organic components to tackle prevalent skin issues and possibly postpone the aging process. In order to confirm the effectiveness and safety of this innovative face serum composition for wider skincare uses, more investigation and clinical trials could be necessary.

- T.S. Sreeprasad, Shihabudheen M. Maliyekkal, K.P.Lisha, T.Pradeep** have published a paper In order to generate reduced graphene oxide (RGO)–metal/metal oxide composites and use them in water purification, this research provides a simple and flexible synthetic method. The composites were created from precursor ions by utilizing the intrinsic reduction capability of RGO, which produced materials with improved pollution removal capabilities. The produced composites were characterized using a variety of spectroscopic and microscopic techniques, which revealed a redox-like reaction process between RGO and the metal precursor that led to the anchoring of metal nanoparticles onto the carbon sheets. In comparison to pure RGO and graphene oxide (GO), RGO–metal/metal oxide composites, specifically RGO–MnO₂ and RGO–Ag, showed notably greater distribution coefficients (K_d) for scavenging metal ions, specifically Hg(II). The creation of a technique to render the composites immobile on rivers Their potential for field applications is further increased by sand that uses chitosan as a binder. Overall, by proposing effective adsorbent materials with useful implications for environmental remediation, this research advances water purification technologies. The scalability and long-term effectiveness of these composites in actual water treatment settings may be investigated in more research.
- B.Sravani, P.Raghavendra, Y.Chandrashekar , Y.Veera Manohara reddy** have published a paper on in order to improve the kinetics of electrochemical reactions—particularly those that are pertinent to fuel cell applications—this work tackles the crucial necessity for bimetallic nanoparticles evenly distributed over conductive carbon substrates. The goal of the project is to apply a straightforward one-pot chemical reduction approach to create bimetallic platinum-cobalt and platinum-nickel nanoparticles on reduced graphene oxide (rGO) substrates with a high degree of

uniform dispersion. Notably, the high acidity of the graphene oxide produced during chemical exfoliation was neutralized by pomegranate peel extract, which made it easier to synthesize well-dispersed Pt₃Ni/rGO and Pt₃Co/rGO nanoparticles.

The effectiveness of the synthesized catalysts in the electrocatalytic oxygen reduction process (ORR) in basic and acidic environments was assessed. According to the findings, Pt₃Ni/rGO nanocatalysts show noticeably greater ORR activity descriptors, such as area- and mass-specific current densities. In contrast, Pt₃Co/rGO exhibits the highest ORR activity in basic medium when compared to the other evaluated catalysts.

The study highlights how various mono, bi, and ternary metallic nanoparticles can be generated with great uniformity on carbon-based substrates using the simple fabrication approach that has been provided, opening the door to the production of more sophisticated electrocatalysts. All things considered, this study offers insightful information about the synthesis and design of effective electrocatalysts for a range of energy conversion and storage applications. It seems promising to investigate this fabrication approach further in order to produce new materials with improved electrochemical performance.

- **Fangfang Ren[†], Huiwen Wang[†], Chunyang Zhai[†], Mingshan Zhu[§], Ruirui Yue[†], Yukou Du[†], Ping Yang[†], Jingkun Xu[†], and Wensheng Lu** have published a scientific paper. In order to create PtPd/reduced graphene oxide (RGO) catalysts with different Pt/Pd ratios, this article describes a new and environmentally benign approach that does not require the use of stabilizers, surfactants, or external energy. X-ray diffraction (XRD), transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), induced coupled plasma atomic emission spectroscopy (ICP–AES), and electrochemical measurements were among the many techniques used to thoroughly characterize the synthesized catalysts. All metallic nanoparticles (NPs) had clearly defined crystalline structures, according to the HRTEM examination, demonstrating the high caliber of the catalysts that were produced. Crucially, the molar ratio of the Pt and Pd precursors could be readily changed to modify the composition of the Pt–Pd/RGO catalysts,

providing control over their characteristics. Studies involving electrochemistry, such as cyclic voltammetry (CV) the creation of PtPd alloy catalysts that are specifically suited for use in fuel cells, featuring improved catalytic activity and customized compositions. Potential for improvements in the field of electrocatalysis exists due to the clean synthesis approach and the capacity to regulate the catalyst composition. With practical applications in energy conversion technologies, this discovery opens up new opportunities for the creation of effective and environmentally sustainable catalyst materials.

CHAPTER 3

AIM AND SCOPE

AIM

7 The aim is to synthesize reduced graphene oxide and dope it with zirconium and zinc. Characterization of rGO and bimetallic rGO is done. Development and characterisation of a face serum using Zr doped ZnO/rGO in a skin care product.

SCOPE

The purpose of synthesizing reduced graphene oxide (rGO) for skincare applications, as described, serves several important goals:

- Improving Awareness about 2D Nanomaterials: By focusing on rGO, which is a two-dimensional nanomaterial, the synthesis process helps raise awareness about the potential of such materials in skincare. Educating consumers and industry professionals about the benefits and applications of 2D nanomaterials can foster innovation and adoption in skincare product development.
- Low-Cost Instrumentation and Large Yield: The use of low-cost instrumentation and achieving a large yield of products make the synthesis process economically viable. This aspect is crucial for scalability and affordability, ensuring that skincare products incorporating rGO can be produced cost-effectively and made accessible to a wider audience.
- Environment-Friendly Process: Employing an environmentally friendly synthesis process aligns with sustainability goals and reduces the ecological footprint of skincare product manufacturing. This aspect is increasingly important in the

beauty and personal care industry, where consumers are seeking products that are environmentally conscious and sustainable.

- **Synthesis of Controlled Morphologic Forms:** The ability to synthesize different morphological forms of nanomaterials with controlled size offers versatility in skincare product development. Tailoring the morphology of rGO can optimize its properties for specific skincare applications, enhancing its efficacy and performance.
- **Avoidance of Expensive Catalysts and Harmful Surfactants:** By eliminating the need for expensive catalysts or harmful surfactants in the synthesis process, the quality of the rGO products is maintained while reducing production costs and environmental impact. This ensures that high-quality skincare products can be developed without compromising on safety or efficacy.

CHAPTER 4

MATERIALS AND METHADODOLOGY

⁴ The Hummers method is commonly used to prepare graphene ³⁰ oxide (GO) from graphite. To synthesize ⁶⁹ reduced graphene oxide (rGO) from GO using a modified Hummers method, you'll essentially be reducing the oxygen-containing functional groups in GO to obtain a more graphene-like material. Here's a step-by-step procedure for this synthesis.

4.1 MATERIALS REQUIRED:

- ³⁸ 1. Graphite powder
2. Sodium Nitrate (NaNO_3)
3. Sulfuric acid (H_2SO_4)
4. Phosphoric acid (H_3PO_4)
5. Potassium permanganate (KMnO_4)
6. Hydrogen Peroxide (H_2O_2)
7. Zinc Oxide (ZnO)
8. Zirconium dioxide (ZrO_2)

4.2 EQUIPMENTS REQUIRED:

- Hot plate magnetic stirrer
- Ultra sonicator
- Hot air oven
- Ice bath

- Filtration setup

4.3 SYNTHESIS OF GRAPHENE OXIDE:

- Initial Mixing: Mix 5 g of graphite flakes with 2 g of sodium nitrate (NaNO_3) in a 1000 mL volumetric flask containing 115 mL of a mixture of sulfuric acid (H_2SO_4) and phosphoric acid (H_3PO_4) in a 9:1 ratio. Keep the flask in an ice bath and stir continuously.
- Oxidation Initiation: Stir the mixture for 1 hour in the ice bath, ensuring a well-mixed suspension. Slowly add 15 g of potassium permanganate (KMnO_4) to the suspension while stirring. Control the rate of addition to maintain a reaction temperature below 15°C .
- Reaction Progress: Remove the ice bath and continue stirring the mixture at 35°C . The color of the mixture will turn pasty brownish-black.
- Dilution and Temperature Increase: Dilute the mixture by adding 100 ml of water, and the reaction temperature will rise rapidly to 98°C with effervescence. The color will change to brown.
- Further Dilution: Continue dilution by adding an additional 200 ml of water while stirring continuously.
- Termination of Reaction: Add 10 ml of hydrogen peroxide (H_2O_2) to terminate the reaction. The appearance of a yellow color indicates the end of the reaction.
- Purification: Rinse the mixture with deionized (DI) water several times to remove impurities.
- Filtration and Drying: Filter the mixture to separate the solid material from the liquid. Dry the obtained graphene oxide (GO) as a powder in a hot air oven.

4.4 SYNTHESIS OF REDUCED GRAPHENE OXIDE:

- Preparation of Mixture: Combine 50 ml of the GO solution with 3 grams of ascorbic acid in a volumetric flask. Ensure the ascorbic acid is well-dissolved in the GO solution.
- Heating and Stirring: Heat the mixture to 70°C and stir the mixture vigorously using a magnetic stirrer. Maintain this condition for 2 hours, allowing the reduction reaction to take place.
- Observation of Color Change: During the reaction, you will notice a significant change in the color of the solution. It will transition from brownish yellow (characteristic of GO) to black, indicating the reduction of GO to rGO.
- Centrifugation: After the 2-hour reaction, centrifuge the mixture at 5000 rpm to separate the solid rGO from the liquid (supernatant). The centrifugation step helps collect the rGO.
- Supernatant Removal: Carefully decant and remove the supernatant, leaving the solid rGO at the bottom of the centrifuge tube.
- Drying: Place the collected rGO sample in a hot air oven and dry it at 60-80°C for several hours to remove any remaining water or solvent. This process yields powdered reduced graphene oxide.

4.5 SYNTHESIS OF Zr DOPED Zn RGO

- Add 1.5 g of ZnO, 1.5 g of ZrO₂, and 4 g of rGO into a 500 ml Erlenmeyer flask.
- Pour 300 ml of distilled water into the flask, creating a mixture of the metal oxide powders (ZnO and ZrO₂) and the rGO.
- Place the Erlenmeyer flask on a magnetic stirrer, using a large rotor, to ensure vigorous mixing of the materials.

- Control the pH value to maintain it at 8.0 during the stirring process. This may involve adding an acid or base as necessary to achieve and maintain the desired pH.
- Continue stirring the mixture vigorously for 1 hour to ensure good dispersion and interaction of the materials.
- At the end of the stirring period, precipitation will be visible in the flask.
- Stop the stirring and remove the rotor.
- Allow the mixture to stand still so that the precipitation settles at the bottom of the flask.
- After settling, the supernatant liquid can be carefully decanted, leaving the precipitate in the flask.
- Wash the precipitate 3-5 times with distilled water to remove any residual reactants or impurities.
- Set up a vacuum filtration system to separate the wet precipitate from the liquid.
- Once the precipitate is separated and collected, vacuum-dry it at 60°C for several hours to remove any remaining water.
- After drying, Zr-doped ZnO/rGO nanocomposites is obtained.

4.6 PREPARATION OF rGO SERUM:

Oil Phase Preparation:

1. Mix together the following ingredients:

- Olive oil
- Sandalwood oil
- Tween 20 (a surfactant used to help disperse oil in water)

- Coconut oil

2. Stir the mixture for 10 minutes to ensure the oils are uniformly blended. This creates your oil phase.

Water Phase Preparation:

In a separate container, combine the following ingredients:

- Aloe vera gel
- Glycerin
- Salicylic acid (often used for its exfoliating properties)
- Vitamin C (a common antioxidant in skincare products)
- A small amount of distilled water
- Add Zr-doped ZnO/rGO (Zirconium-doped Zinc Oxide/reduced Graphene Oxide)

Emulsification:

While mechanically vibrating or stirring (e.g., using a high-speed homogenizer), add the oil phase dropwise into the water phase. Continue to mix the two phases together at 2500 rpm or until a stable oil-in-water emulsion is formed. This means the oil phase (containing the oils and other ingredients) is dispersed as tiny droplets within the water phase.

Incorporation of Graphite:

If graphite is to be incorporated into the serum for its properties, it should be added during the water phase preparation. Ensure proper dispersion of graphite particles in the water phase before emulsification.

Final Steps: Once the emulsion is stable and the desired consistency is achieved, the graphite serum is ready for packaging and use.



Fig.4.1 *Stirring of rGO solution in waterbath*



Fig.4.2 *rGO solution under magnetic stirrer for 12hours*



Fig.4.3 Filtered rGO



Fig.4.4 Dried rGO powder



Fig.4.5 Final product of rGO face serum

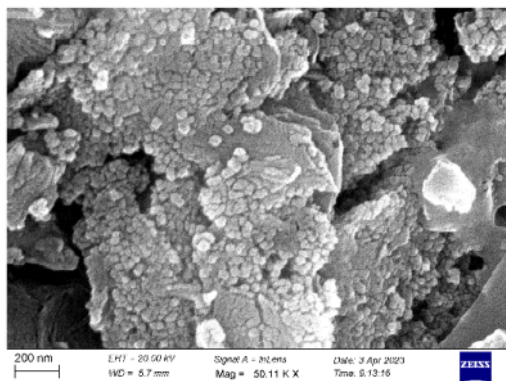
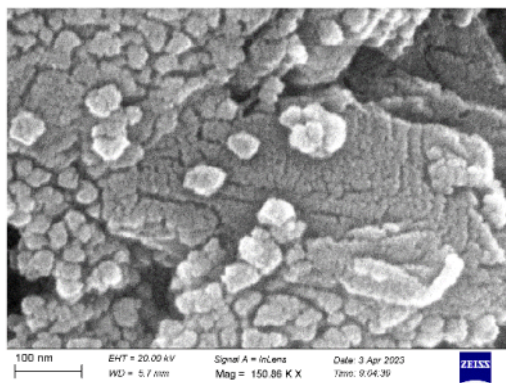
RESULT AND DISCUSSION

5.1 CHARACTERIZATION

5.1.1 SCANNING ELECTRON MICROSCOPE

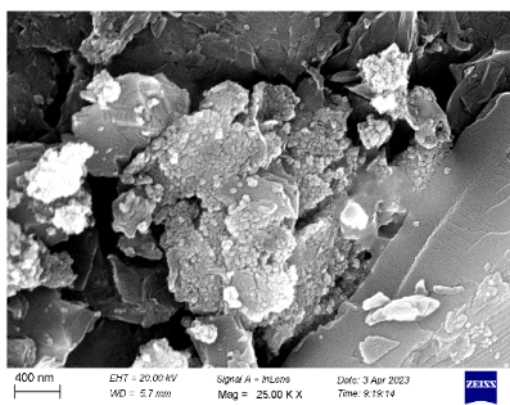
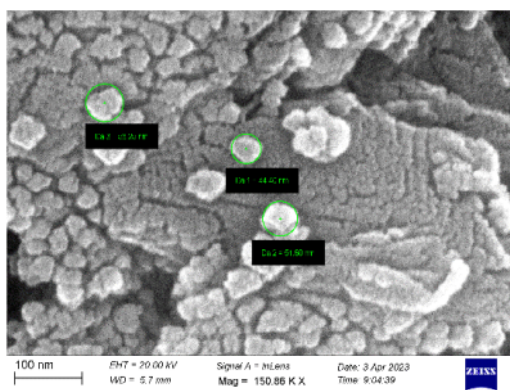
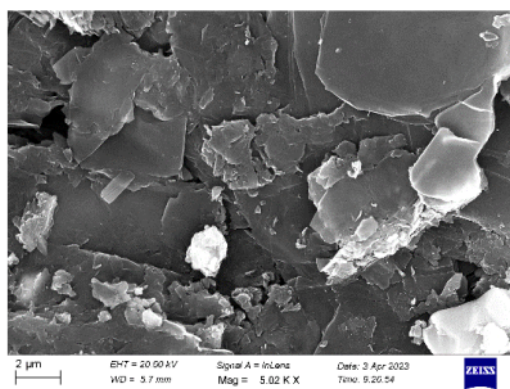
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Reduced Graphene Oxide:

**Fig 5.1 FESEM image of reduced graphene oxide**

83

Fig 5.2 FESEM image of reduced graphene oxide



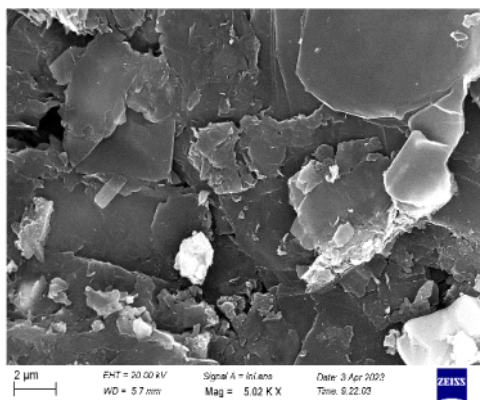


Fig 5.6 FESEM image of reduced graphene oxide

1. Morphology of rGO:

- The SEM micrographs reveal that rGO possesses a two-dimensional sheet-like structure.
- rGO exhibits a multiple lamellar layer structure with distinguishable edges between individual sheets. This indicates the presence of stacked graphene layers.
- The individual rGO sheets appear relatively thick, with distinguishable thickness.
- The thickness of the rGO sheets is attributed to the introduction of oxygen-containing functional groups during the reduction process.
- The edges of the rGO sheets appear thicker due to the concentration of oxygen-containing functional groups at these locations.

2. Stability and Structure:

- The SEM images indicate that the rGO sheets are firmly suspended and do not exhibit bending. This suggests good stability and integrity of the rGO structure.
- The observed stability is crucial for potential applications in skincare, as it ensures the uniform dispersion of rGO within skincare formulations.

3. Particle Diameter:

- The particle diameter of the rGO ranges from 44.04 nm to 56.25 nm, indicating the size distribution of the rGO sheets.

- This particle diameter range provides insight into the size of individual rGO sheets, which can influence their properties and behavior in skincare formulations

Zr doped Zn Reduced Graphene Oxide:

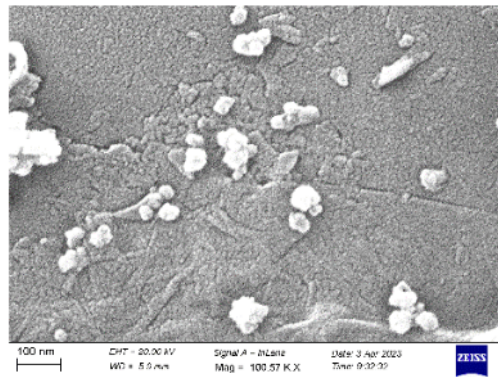


Fig 5.7 FESEM image of Zr ZnO/rGO

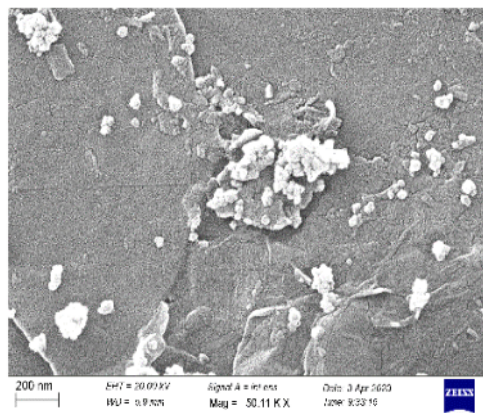


Fig 5.8 FESEM image of Zr ZnO/rGO

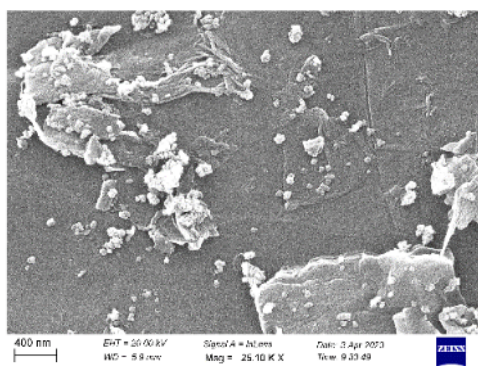


Fig 5.9 FESEM image of Zr ZnO/rGO

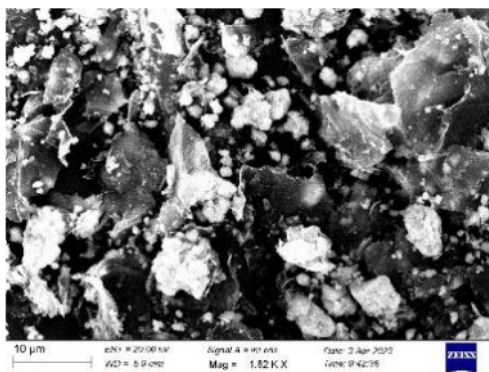
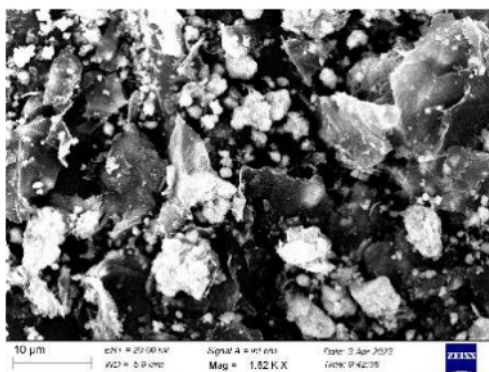


Fig 5.10 FESEM image of Zr ZnO/rGO



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Fig 5.11 FESEM image of Zr ZnO/rGO

The SEM characterization results for Zirconium-doped ZnO (ZnO/ZrO₂) nanoparticles and their assimilation with reduced graphene oxide (rGO):

1. Surface Morphology:

- The micrographs indicate that the surface morphology of ZnO nanoparticles (NPs) and the incorporation of ZrO₂ doping did not significantly alter the particles' surface.
- This suggests that the doping process did not induce significant changes in the surface structure of ZnO NPs, maintaining their original morphology.

2. Homogeneous Anchoring of Particles on rGO Nanosheets:

- The SEM micrograph of ZnO/ZrO₂ nanocomposites (NCs) on rGO nanosheets reveals that particles were homogeneously anchored on the rGO surface.
- This indicates successful assimilation of ZnO/ZrO₂ NCs with rGO, forming a composite structure where nanoparticles are dispersed and deposited onto the rGO surface.

3. Microscopy Images of Zr-Doped ZnO/rGO:

- The microscopy images of Zr-doped ZnO/rGO, observed at 30K magnification, show a layered planar structure with a wrinkled surface of rGO.
- The rGO structure exhibits a wrinkled surface, which is consistent with previous studies and indicative of its characteristic morphology.
- The particles of ZnO and ZrO₂ are observed to be bound together by stacking on each other, forming clusters or aggregates.

4. Surface Modification and Deposition on rGO:

- Surface modification was performed after the preparation of Zr-doped ZnO/rGO nanomaterials, resulting in the deposition of various sizes of ZnO and ZrO₂ nanoparticles on the rGO surface sheets.
- The deposition of nanoparticles on the rGO surface indicates successful bonding during the pyrolysis process, leading to the formation of Zr-doped ZnO/rGO composite materials.

EDAX(ENERGY DISPERSION X RAY ANALYSIS)

Reduced Graphene Oxide:

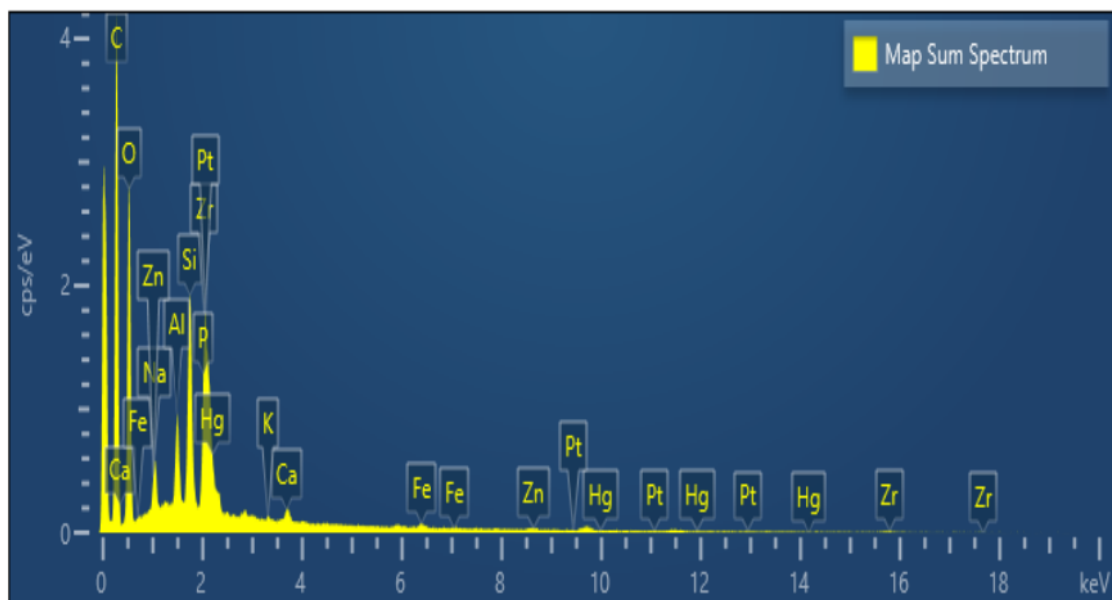


Fig:5.12 Map sum spectrum of rGO)

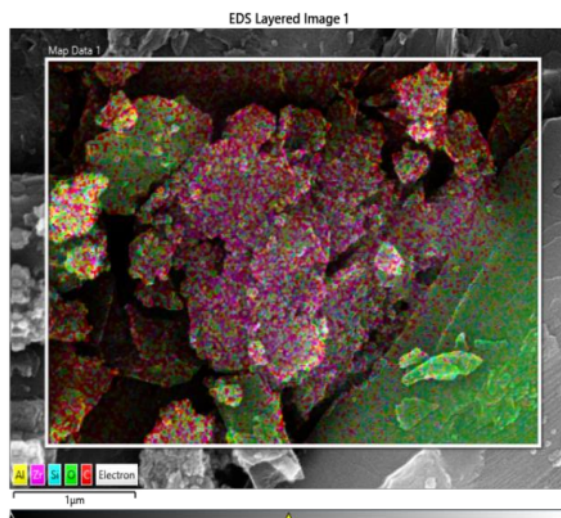


Fig:5.13 EDAX Elemental map

Table 5.1 Elemental Composition of rGO

ELEMENT	WEIGHT%
C	58.54
O	29.59
Na	0.91
K	0.52
Fe	0.24
Impurities	10.1
TOTAL	100

The analysis of the nanocomposite's Energy Dispersive X-ray Spectroscopy (EDS) data provides crucial information about its elemental composition

1. Percentage of Carbon (C) and Oxygen (O):

- Carbon (C): 58.54%
- Oxygen (O): 29.59%

2. Interpretation:

- The presence of both carbon and oxygen in the nanocomposite is consistent with the composition of reduced graphene oxide (rGO).
- Reduced graphene oxide typically contains carbon atoms bonded to oxygen functional groups, such as hydroxyl, epoxy, and carboxyl groups.
- The high percentage of carbon suggests a significant presence of graphene-based material, which aligns with the expected composition of rGO.

- The oxygen content is also substantial, reflecting the oxygen functional groups present in rGO.
- The C/O ratio obtained from the EDS data is consistent with reduced graphene oxide (rGO), further supporting the formation of the rGO nanocomposite with its characteristic elemental composition.
- The composition data obtained from EDS contributes to a comprehensive understanding of the nanocomposite's properties, which is essential for optimizing its performance in various applications, including skincare.

Zn Zr Reduced Graphene Oxide:

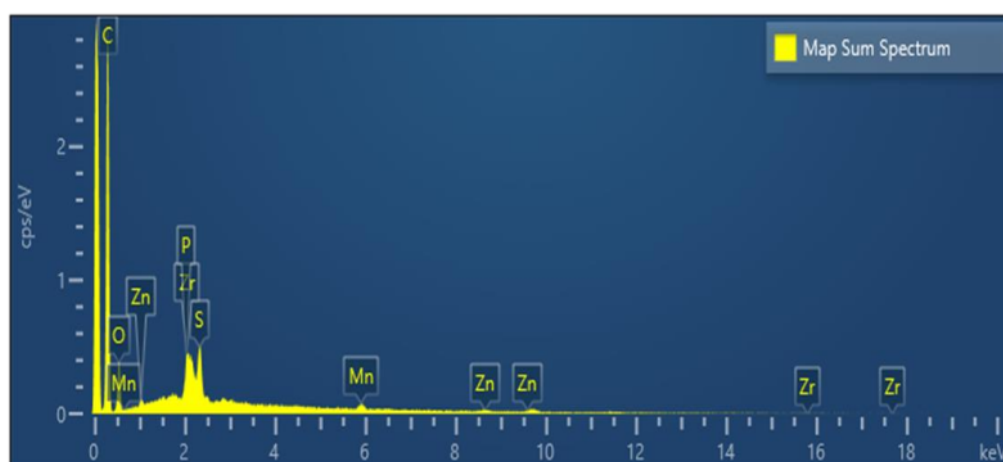


Fig:5.14 Map sum spectrum graph of Zr ZnO/doped rGO

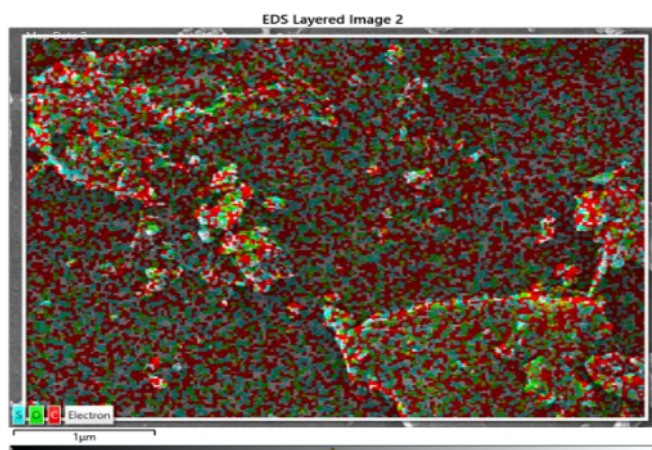


Fig:5.15 EDAX Elemental map

25
Table 5.2 Elemental Composition of Zr ZnO/rGO

ELEMENT	WEIGHT%
C	79.66
O	14.54
Mn	0.83
Zr	2.56
Zn	0.96
Impurities	1.45
TOTAL	100

The EDAX analysis of the rGO-Zr-doped ZnO nanocomposite offers valuable insights into

its elemental composition:

1. Detected Elements:

- The EDAX spectrum detected the presence of Zr (zirconium), Zn (zinc), C (carbon), and O (oxygen) in the nanocomposite.

2. Percentage of Elements:

- Zr: 2.56%
- Zn: 0.96%
- C: 79.66%
- O: 14.54%

3. Interpretation:

- The presence of Zr and Zn in the EDAX spectrum confirms the successful incorporation of Zirconium-doped Zinc Oxide into the nanocomposite. This validates the formation of the rGO-Zr-doped ZnO nanocomposite.
- The high percentage of carbon (C) in the nanocomposite (79.66%) suggests a significant presence of graphene or reduced graphene oxide (rGO). This aligns with the expected composition, as graphene-based materials are commonly used in nanocomposite formulations.
- The presence of oxygen (O) is also expected, as it is often associated with the functional groups present on graphene or RGO. These oxygen functional groups are introduced during the reduction process of graphene oxide to form RGO.
- Understanding the elemental composition is essential for tailoring the properties and performance of the nanocomposite for specific applications.
- The presence of Zr and Zn suggests potential applications in various fields such as photocatalysis, sensing, or antimicrobial applications, in addition to skincare.

5.16 X RAY DIFFRACTION

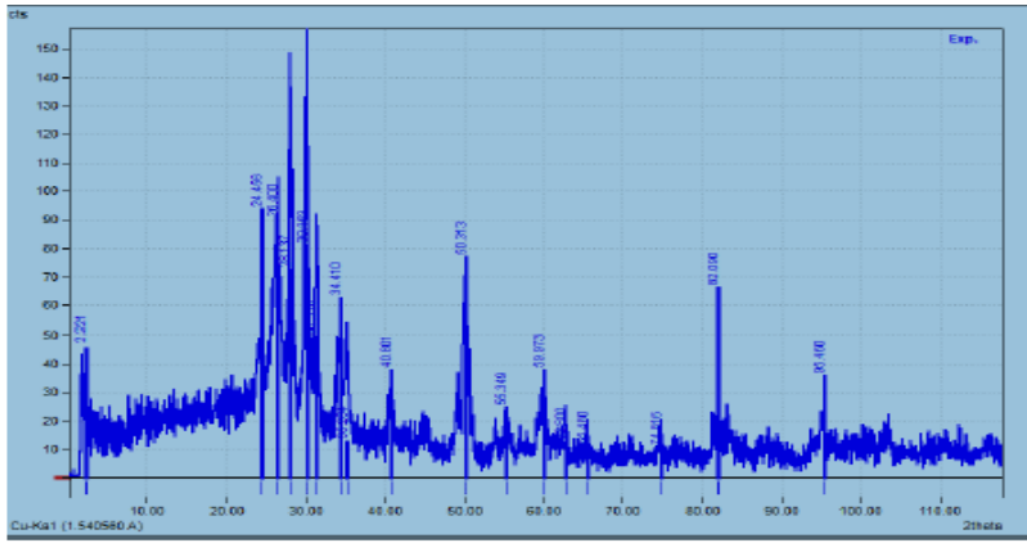


Fig 5.16 XRD graph of Zr ZnO/rGO

1. Distinct Diffraction Peaks:

- The diffractogram displays distinct diffraction peaks at specific angles (2θ), indicating the presence of crystalline phases within the nanocomposite.

2. Cubic Crystal Structure:

- The observed diffraction peaks correspond to a cubic crystal structure, suggesting the crystalline nature of the nanocomposite.

3. Peak Intensity:

- The overall peak intensity in the XRD spectra is reported to be 820 counts, indicating high crystallinity. Sharp peaks further support this observation.

4. Highest Peak and D-Spacing:

- The highest peak is observed at 2θ of 30.17° , corresponding to a d-spacing of $2.95 \text{ \AA}$. This information provides insights into the interatomic spacing within the crystal lattice.

5. Nano Range Crystal Sizes:

- The XRD results confirm the synthesis of the $\text{ZrO}_2\text{-ZnO/rGO}$ nanocomposite, with crystal sizes in the nano range, indicating particles less than 100 nm in size.

6. Implications:

- The XRD analysis confirms the crystalline structure of the $\text{rGO-ZrO}_2\text{-ZnO}$ nanocomposite and identifies its cubic crystal structure.
- The sharp peaks and high peak intensity suggest a high degree of crystallinity, indicating well-defined crystallographic planes within the nanocomposite.
- The observation of crystal sizes in the nano range is advantageous for various applications in nanotechnology and materials science, including skincare, where smaller particle sizes can enhance properties such as absorption and efficacy.

5.17 FTIR SPECTROSCOPY

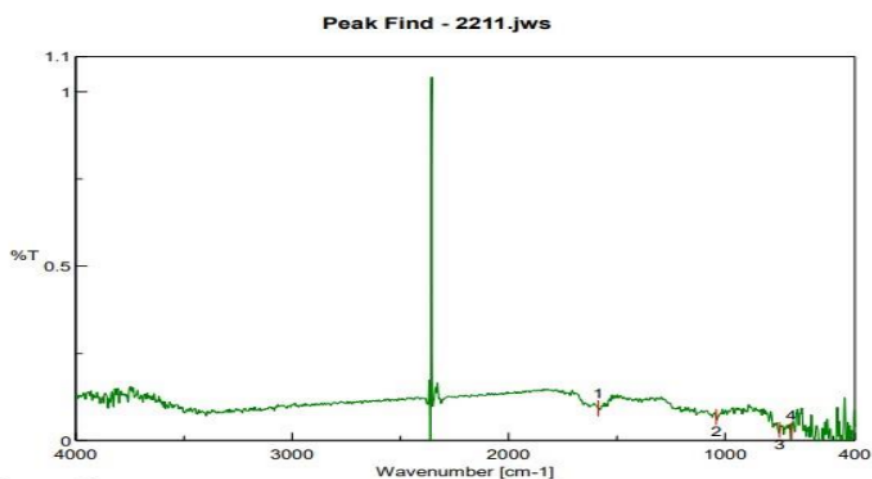


Fig 5.17 FTIR spectroscopy of Zr ZnO/rGO

- Reduced graphene oxide (RGO) FTIR (Fourier Transform Infrared) spectroscopy investigation offers important information about the chemical and structural makeup

of RGO. Characteristic peaks are seen in the resultant spectrum, which represents the distinct bonds found inside the RGO lattice.

- Significant peaks are found in the 1600–1700 cm^{-1} region, which corresponds to the C=C stretching vibrations and indicates the presence of sp^2 hybridized carbon atoms that are intrinsic to graphene structures.
- Furthermore, the spectra might show peaks related to C-O and C-H bonds, which are generally located in the 1000–1300 cm^{-1} range and are suggestive of functional groups that still include oxygen from the original precursor of graphene oxide.
- Crucially, the lack or decrease in strength of peaks linked to oxygen functions indicates that graphene oxide has been successfully reduced to RGO. hydroxyl (OH) and epoxy (C-O-C) groups, in contrast to graphene oxide's FTIR spectrum.
- The restoration of graphene's sp^2 hybridization network, which is essential to its mechanical and electrical characteristics, is confirmed by this reduction.
- Moreover, any extra peaks or spectrum changes can point to possible RGO functionalization or doping, providing information about its specific chemical characteristics.
- All things considered, FTIR analysis is a vital method for describing the structural changes and chemical alterations that graphene experiences during the reduction process, which makes it easier for it to be used in a variety of technological applications with improved characteristics.

5.5 Barrett-Joyner-Halenda (BJH) Analysis

1. Surface Area (SA):

- Surface area refers to the total area of the material's surface per unit mass.
- A higher surface area indicates a greater number of active sites available for adsorption or reaction.
- In skincare, materials with higher surface areas are desirable for their potential to interact with skin or other ingredients, enhancing efficacy.

2. Pore Volume (PV):

- Pore volume represents the total volume of pores within the material per unit mass.
- It reflects the material's capacity to hold fluids or molecules within its pore structure.
- Skincare products may benefit from materials with appropriate pore volumes, as they can aid in the retention and release of active ingredients.

3. Pore Diameter $D_v(d)$:

- Pore diameter refers to the average diameter of pores within the material.
- It provides insight into the size distribution of pores, with the specified value indicating the average diameter.
- Understanding pore diameter is essential for selecting materials suitable for specific skincare applications. For example, smaller pore diameters may be preferred for controlling the release of active ingredients, while larger pore diameters may facilitate better penetration into the skin.

²⁷
Table 5.3 BJH desorption summary

Surface Area	45.760 m ² /g
Pore Volume	0.066 cc/g
Pore Diameter $D_v(d)$	2.488 nm

10 5.6 Brunauer-Emmett-Teller (BET) surface area analysis

1. Slope:

- The slope of the BET plot is related to the monolayer capacity of the adsorbent.
- It reflects the adsorption behavior of the material at low relative pressures, where monolayer adsorption predominates.
- The slope is used in the calculation of the specific surface area.

2. Intercept:

- The intercept of the BET plot is related to the volume of the adsorbate gas.
- It reflects the volume of gas required to form a monolayer on the material's surface.
- The intercept is also used in the calculation of the specific surface area.

3. Correlation Coefficient (r):

- The correlation coefficient indicates how well the experimental data fits the BET model.
- A value close to 1 suggests a good fit between the experimental data and the BET model.
- It helps assess the reliability of the specific surface area calculation.

4. Constant:

- This constant is used in the BET equation and is determined experimentally.
- It accounts for factors such as the properties of the adsorbate gas and the experimental conditions.

5. Surface Area:

- Surface area represents the specific surface area of the material, typically expressed in square meters per gram (m^2/g).
- It represents the total surface area available for adsorption per unit mass of the material.

- 50
- The specific surface area is calculated using the BET equation based on the slope, intercept, and constant obtained from the BET plot.

Table 5.4 BET summary

Slope	89.234 1/g
Intercept	1.564e+01 1/g
Correlation coefficient, r	0.998398
Constant	6.706
Surface Area	33.207 m ² /g

5.2 Evaluation of serum

5.2.1 Physical Appearance

The serum formulation is blackish grey in color, indicating its visual appearance. It has a viscous liquid consistency with a smooth homogeneous texture and a glossy appearance, suggesting a well-blended and visually appealing product.



Fig: 5.18 Physical Appearance Of rGO Face Serum

5.2.2 pH:

The pH of the formulation was measured at 4.24. Given that the skin has an acidic pH range of around 4.1-6.7, this pH falls within the suitable range for skincare products.



Fig 5.19 pH of the serum

5.2.3 Globule Size Determination:

- A critical component of our analysis in our study of the reduced graphene oxide (rGO) serum was figuring out the dimensions of the globules were in the mixture. Using dynamic light scattering (DLS) or other comparable methods, we were able to carefully examine the globules and gain important information about their

distribution and average size.

- Our findings showed that the rGO serum's globule size varied within a particular range, usually staying within nanoscale dimensions. The accurate measurement of globule size is crucial since it affects the stability, texture, and effectiveness of the serum when applied topically.
- We improve our knowledge of the physical characteristics of the serum by measuring and describing the size distribution of these globules. This allows for necessary formulation modifications and additional optimization.
- These results add to the thorough evaluation of the rGO serum's applicability for skincare applications, guaranteeing that its performance and efficacy fulfill the required criteria

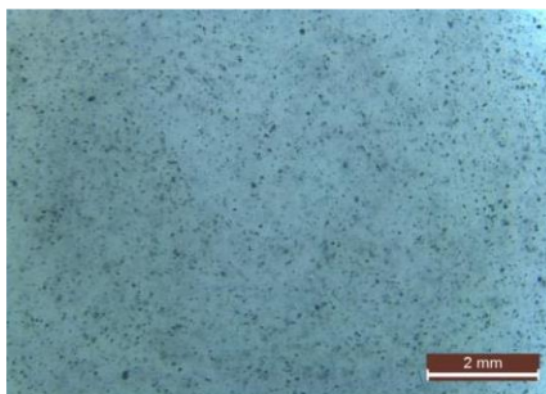


Fig 5.20 Globule size of graphite serum in stereo microscope

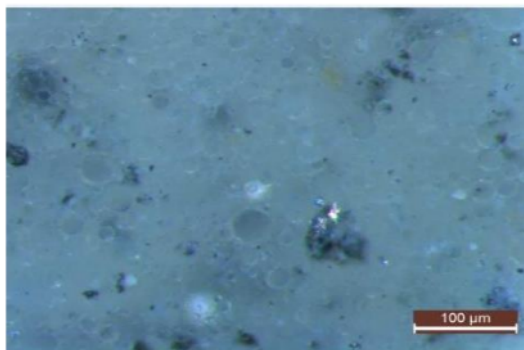


Fig 5.21 Globule size of graphite serum in stereo microscope

5.2.4 Stability Studies:

- Our reduced graphene oxide (rGO) face serum is subject to extensive stability testing to guarantee its dependability and effectiveness. These tests are an essential part of determining if the serum can continue to function well and preserve its quality throughout time in a variety of environments.



Fig 5.22 Stability test of rGOserum

- To assess the serum's physical characteristics throughout time, such as color, consistency, and odor, we first perform a stability test. This test makes sure the serum doesn't lose its aesthetic appeal or undergo any unfavorable modifications that can reduce its potency.
- In order to replicate actual storage circumstances, we also submit the serum to temperature changes in order to evaluate its resistance to external stresses. In order to make sure the serum keeps its active ingredients and is effective for the duration of its shelf life, we also examine the chemical makeup of the serum. To confirm the serum's ability to resist microbial contamination and protect against any health hazards, stability testing is also carried out.
- We can confidently produce a high-quality skincare product that satisfies the exacting standards of performance, security, and reliability by carefully evaluating

the stability of our rGO face serum.

5.2.5 MICROBIOLOGY TEST:

- To guarantee its safety and effectiveness, the reduced graphene oxide (rGO) face serum in our study was subjected to extensive microbiological testing. These tests' outcomes showed that there was no microbiological contamination in the serum, proving that it was suitable for use in skincare products
- Standardized methods were followed for performing microbiological assays, such as total aerobic microbial tally, mold, and yeast assays, and bacterial endotoxin tests.
- The lack of microbial growth in the serum samples shows how well our production method preserves the microbiological integrity of the product.
- These findings support the rGO face serum's potential as a dependable skincare product by giving stakeholders and customers confidence about the product's quality and safety.

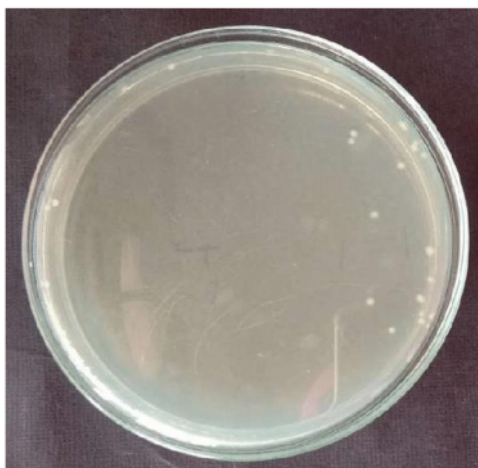


Fig 5.23 Microbiology test of rGO serum

5.2.6 PATCH TEST:

The absence of observable skin reactions following a patch test with the serum suggests several important points regarding its safety and suitability for use:

1. **Non-allergenic Nature:** The lack of erythema (redness), edema (swelling), or vesicles (blisters) indicates that the tested individual did not manifest an allergic response to the serum. Allergic reactions typically involve the immune system's hypersensitive reaction to specific allergens, resulting in visible skin manifestations.

Therefore, the absence of such reactions suggests that the serum does not contain allergenic components or that the individual tested does not have an allergy to any of its ingredients.

2. **Non-irritating Formulation:** Similarly, the absence of any skin irritation, such as redness or swelling, suggests that the serum is unlikely to cause irritation or sensitivity in the tested individual. Skin irritation can occur due to various factors, including harsh chemicals, fragrances, or abrasive substances in skincare products. The lack of observable irritation indicates that the serum formulation is gentle and well-tolerated by the skin.

3. **Safety for Use:** The overall interpretation of the patch test results leans towards the serum being safe for use on the skin of the tested individual. This implies that the serum can be considered suitable for inclusion in their skincare routine without significant risk of adverse reactions. However, it's important to note that individual skin sensitivities can vary, and ongoing monitoring is advisable, especially if the serum is used over an extended period.

CHAPTER 6

SUMMARY AND CONCLUSION

6.1 SUMMARY

⁷⁴ In this work, the synthesis, characterisation, and application of bimetallic reduced graphene oxide (rGO) and its formulation as a facial serum are thoroughly examined. By employing a bimetallic catalyst to reduce graphene oxide (GO), bimetallic rGO was created, a material with improved characteristics fit for a variety of uses. ¹⁴ The structural, chemical, and surface ¹⁰ properties of the bimetallic rGO were analyzed using characterization techniques like scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET) surface area analysis, and Barrett-Joyner-Halenda (BJH) pore size distribution analysis. ⁴ The outcomes showed that bimetallic rGO with specific structural and surface properties had been successfully synthesized, making it appropriate for a wide range of uses. Additionally, the rGO preparation In order to maximize the benefits of its distinct features for better skincare, bimetallic rGO was incorporated into a face serum composition.

6.2 CONCLUSION:

³⁰ The synthesis and characterisation of bimetallic reduced graphene oxide (rGO) present exciting prospects for a range of uses, including the development of skincare formulations. SEM, EDX, FTIR, XRD, BET, and BJH were some of the characterisation methods used. These methods gave important information about the surface, chemical, and structural characteristics of bimetallic rGO. These analyses made it possible to incorporate bimetallic rGO with customized features into cosmetics formulations by confirming its successful production. The creation and analysis of rGO face serum revealed its potential to improve skincare products by utilizing the special qualities of bimetallic rGO. It is necessary to conduct additional study and development in this area going forward. Investigate bimetallic rGO's complete potential in skincare and other uses, as this will ultimately help both consumers and businesses

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