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RESEARCH ARTICLE

GREEN SYNTHESIS OF *ANNONA MURICATA* (L) ZNO NPS UV ABSORBER ON TEXTILE.

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Abstract

This present study to develop an UV absorber ZnO NPs prepared by green method (GM) using *Annona muricata* (L) leave extract and chemical method (CM) using zinc acetate and sodium hydroxide as a substitute for the chemical method. The characterization of ZnO NPs confirmed by Ultraviolet Visible Spectroscopy, XRD, SEM, EDAX, FTIR and. UV-absorbers (ZnO NPs) coated on cotton bleached fabric were evaluated the Ultraviolet Protection Factor it showed the mean UPF 30.26 and 24.58 good and very good protection level according to Australian/New Zealand Standard (AS/NZS 4399) that revealed the significant improvement in the UV absorbing properties on treated cotton fabric

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Introduction:-

UV absorbers are a substance to capable of absorbing short wave solar radiation and of restoring the absorbed energy to the environment and decrease the harmful effects of UV radiation on human skin, as well as the textile materials. [1,2] The standard rate of UV protection, is depending on numerous factors, such as weave of the material and chemical additives such as UV absorbers. [3] It has significant impact on the UV protection properties, there are various UV absorbers frequently added to the textile during manufacturing process. [4] ZnO is one of the inorganic UV absorber; the mechanism of UV absorption is semiconductor material involves the use of photon energy to excite electrons from the valence band to the conduction band. [5] The bandgap energy (ZnO =3.37 eV), which is highly depending on the degree and the kind of crystalline of the inorganic coating. [6] Excellent light fastness and UV protection has much longer period than organic UV absorbers, [5] cost effective, high yielding and simple to implement. [7]

ZnO has been obtain by precipitation in an emulsion system with zinc acetate dehydrate used as a precursor of potassium hydroxide or sodium hydroxide as precipitate agent. [8] These chemical approach productions can lead to the development of inorganic nanoparticles with large scale of fabrication. [9] There are a several essential aspects of synthesis with environmental concerns, with involving the selection of nontoxic, capping and reducing agents, the choice of harmless solvents and the development of energy accomplished using green method. [10] Green chemistry can build into a moderately pollutant free chemicals and solvents water, natural extracts from plants aqueous leaf extracts. [11]

Natural substances such as phenolic acids, flavonoids, anthraquinones and high molecular polyphenols have been considered as sunscreen agents because of their ultraviolet ray absorption in the UV region. [12,13] Plant extract contains polyol compounds which are responsible for the reduction of met ions and water soluble heterocyclic components stabilize the metal nanoparticles formed. This reduction of the metal salts and formation of

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nanoparticles using plant extract mainly depends upon the choice of solvent reducing agent and the capping agent used. [14,15] Plant extracts containing bioactive alkaloids, phenolic acids, poly phenols, proteins, sugars and terpenoids are have an important role in first reducing the metallic ions and then stabilizing. [16] In general the plant chemical that protect plant cells from environmental hazards such as pollution, stress, drought, UV exposure and pathogenic attack. [17,18] The absorption of the secondary metabolites phenolic, glucoside, phenolic acids, favonoids. [19] UVB absorbing compounds other than Flavonoids, some phenolic acids (Singapyl esters) and carotenoids have recently been acknowledge to play a role in UV protection. [20]

In the case of every individual plant responses specific UV protective and adaptive have been found induction of UV absorbing phenolic compounds that afford UVB attenuation. [21,22] UVB radiation leads to increase in the amount of Flavonoids and phenolic acids [19,23] UVB attenuation, depending on their leaf structure and pigment composition, variation in susceptibility to enhance. [19,24] UVB induced accumulation of UVB absorbing compounds. [24,25]

Annona muricata (L), commonly known as soursop, graviola, a native to the warmest tropical area in South and North America and is now widely distributed throughout tropical and subtropical parts of the world India, Malaysia and Nigeria. [26] *A. muricata* is an evergreen, erect tree reaching 5-8 m in height. [27] *Annona* leaf family annonaceae, Tamil mulluchitta, [28] it is a medicinal herb is extensively accessible in India, these plant leaves, stem; root and seed all the parts are having antibacterial activity against numerous pathogens, Antitumor, Anti- parasitic, Insecticidal and Anti-microbial activities. [29,30] The extracted from its leaves treat with various skin disorders. [31] The presents on phytochemicals are alkaloids, Flavonoids, carbohydrates, cardiac glycosides, saponins, tannins, phytosterol, terpenoids and proteins. [32]

In the present work deal with the synthesis and characterization of UV absorber (ZnO NPs) Green and chemical method particles and their characterization were analysed and applied on to cotton bleached fabrics using pad-dry-cure method to evaluate the UV protection function in the treated textiles by standardized test procedures.

Material and Methods:-

Selection of the Material: 100% grey cotton 40s count fabric (purchased from Xavier fabrics Madurai, Tamil Nadu, India.) pre-treated, bleached fabric were selected for the further study.

Selection of Chemicals: Zinc acetate dehydrate (Merck, 99%), Sodium hydroxide (NaOH) (Merck, 99%) was used as the introductory material was supplied by Sigma-Aldrich chemicals.

Plant Source: Fig 1 shown the *Annona Muricata* (L) plant leaves were collected Vettoornimadam, Nagercoil, Kanyakumari District, Tamil Nadu.



Fig 1:- *Annona Muricata* (L) Leaves

Scientific Classification of *Annona Muricata* (L)

Kingdom : **Plantae**
Order : **Magnoliids**

Family : Annonaceae
 Genus : *Annona*
 Species : *A. muricata*

Table 1:- Phytochemical Screening of *Annona muricata* (L) ethanolic leaf extract

Flavonoide	+ve
Saponins	+ve
Tannins	+ve
Steroids	-ve
Alkaloids	+ve
Triterpenoids	+ve
Anthrequinone	-ve
Reducing sugar	-ve
Cardiac glycosides	+ve

Where +ve = Present, -ve- absent [30]

Qualitative phytochemical of *Annona muricata* (L) leaves extract contain Flavonoids, polypehenol, phobattanins, tannin, saponins glycosides, anthroquinones, polyphenol, steroids, alkaloids, carbohydrate, amino acid and steroids. Quantitative analysis revealed that *Annona muricata* (L) rich amount of total phenol (178.61 ± 1.45 mg/gm), tannis (20.08 ± 0.25 mg/gm), alkaloids (85.23 ± 0.38 mg/gm), saponin (35.59 ± 0.18 mg/gm), and Flavonoids (121.09 ± 0.62 mg/gm), were presented. [28]

Preparation of plant Extraction:-

Annona muricata (L) fresh leaves were washed double distilled water in order to remove the dust particles and air dried for 15 days and to make a fine powder in an electric grinder. The plant extract was prepared by mixing 50 g of leaf powder with 500 mL of ethanol solvent extract with constant stirring on a magnetic stirrer. Filtered the extract through a Whatman no. 1 filter paper, and the filtrate was stored at -4 °C temperature.

Synthesis Procedure:-

ZnO NPs was synthesised by two methods, Green Method-(GM) and Chemical Method-(CM).

Methods Flow Chart

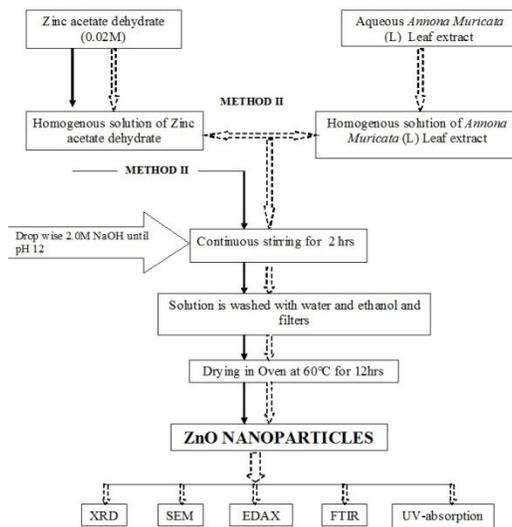


Fig 2:- Synthesis process GM and CM

Green Method (GM):-

50 ml of ethanol 0.02M aqueous Zinc acetate dehydrate was added under constant stirring at 60°C. Aqueous leaf extract of *Annona Muricata* (L) were introduced into the above solution after 20 min stirring at different sets (0.25, 0.5, 1 ml). To the same 2.0M NaOH was added to make pH 12 resulted in a pale white aqueous solution. This was

then placed in a magnetic stirrer for 2hrs. The resultant the solution was allowed to settle for overnight and the supernatant solution was discarded carefully. The remaining solution was centrifuged at 5000 rpm for 20 min and then taken out and washed over and over again with distilled water followed by ethanol. The obtained powder dried at 60°C for 12hrs in hot air oven, to get the zinc oxide nanoparticles.

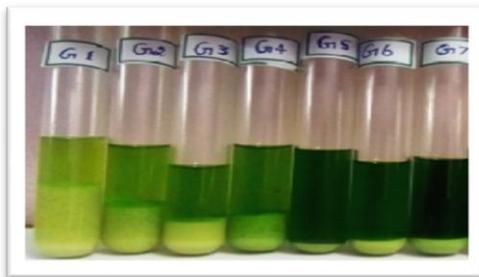


Fig 3:- Green Method (GM) synthesis optimizing concentration

Fig 2 shown the similar process follow by the entire Green Method synthesis process optimize with different concentration of *Annona muricata* (L) leave extract eventually named as G1-1:1, G2- 1:2, G3-1:3, G4-1:4, G5-1:5, G6-1:6, G7-1:7. For example G1 1:1 (Zinc acetate dehydrate : *Annona muricata* (L) leave extract), temperature and concentration involving the interplay between the metal ion precursors and the reducing agent.[33]

Chemical Method (CM):-

0.02M aqueous Zinc acetate dehydrate was dissolved in 50ml ethanol under vigorous stirring at 60°C, aqueous 2.0M NaOH was added drop by drop to reach pH 12. This was then placed in a magnetic stirrer for 2hrs. After completion of the reaction, the white precipitate formed was centrifuged at 5000 rpm for 20 min then taken out and washed thoroughly with distilled water followed by ethanol to remove the impurities. The precipitate was dried in a hot air oven at 60°C for 12hrs.

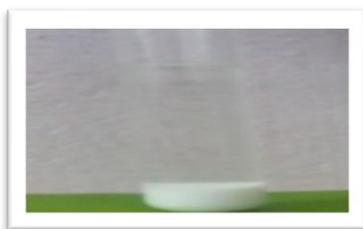


Fig 4:- Chemical Method (CM)

UV absorber Finish on Fabric:-

UV absorber ZnO NPs coated bleached cotton fabric by pad-dry-cure method. These UV absorber nanoparticles have a primary role in determining their adhesion to the fabrics. 1% of ZnO NPs GM and CM UV absorbers were coated 1:20 material liquor ratio with 1% of acrylic binder. Nanoparticles are well bound to the fabric surface even without the use of a binder. However, a binder may used, when a higher level of wash fastness is required. [34] To the coating process padding mangle speed was 15 m/min with a pressure of 15 kg/cm² were followed, after padded fabrics were taken out dried naturally and cured for 3 minutes at 150°C.

UV absorber (ZnO NPs) Characterization Analysis:-

The Phase evolution of calcined powder as well as that of sintered samples was studied by X-ray diffraction (XRD) technique (Panalytical X-ray Diffractor) using Cu K α radiation. The generator voltage and current was set at 40 KV and 30 mA respectively. Samples were scanned in the 2 θ range in continuous scan mode. The scan rate was 0.04o/sec. All the peaks can be well indexed to the Zincite phase of ZnO (International Center of Diffraction Data JCPDS) Particle diameter D was calculated via the Debye-sherrer formula $D = \frac{K\lambda}{\beta \cos\theta}$, where K is Sherrer constant, λ is the X-ray wavelength (1.54060 Å), β is the peak width of half maximum, and θ is the Bragg diffraction angle. Scanning Electron Microscopy (SEM) VEGA 3 TESCAN machine was used to characterize the particle size, morphology of nanoparticles. EDAX measurements were confirm the structural element analysis carried out by X-Flash-Bruker. The characterization involved Fourier Transform Infrared Spectroscopy (FTIR) analysis of nanoparticles by Perkin in Elmer Spectrum 1000 spectrum in attenuated total reflection mode, and using

the spectral range 4000-400 Cm^{-1} with the resolution of 4 cm^{-1} . UV absorption were characterised in Perkin Elmer UV-VIS Spectrometer, Lamda -19 to know the kinetic behaviour of nanoparticles. The scanning range for the samples was 200-800 nm at a scan speed of 480 mm/min. The spectrophotometer was equipped with 'UV Winlab' software to record and analyze data. The UV-Vis absorption spectra of all the samples were recorded and numerical data were plotted.

Ultraviolet Protection Factor:-

UPF categories with relative transmittance and protection level equation (1) according to the Australian/New Zealand Standard (AS/NZS 4399). It was the first among the existing classification systems and is the most widely adopted it established a classification system of fabrics according to their sun protection properties shown in table2. The results are discussed in respect to this standard. [35]

$$UPF = \sum_{290}^{400} \frac{E(\lambda) \cdot S(\lambda) \cdot \Delta(\lambda)}{E(\lambda) \cdot T(\lambda) \cdot S(\lambda) \cdot \Delta(\lambda)} \quad (1)$$

Where

$E\lambda$ = erythermal spectral effectiveness,

$S\lambda$ = solar spectral irradiancies $\text{Wm}^{-2}\text{nm}^{-1}$,

$T\lambda$ = spectral transmittance of the fabric,

$\Delta\lambda$ = the bandwidth in nm,

λ = the wavelength in nm.

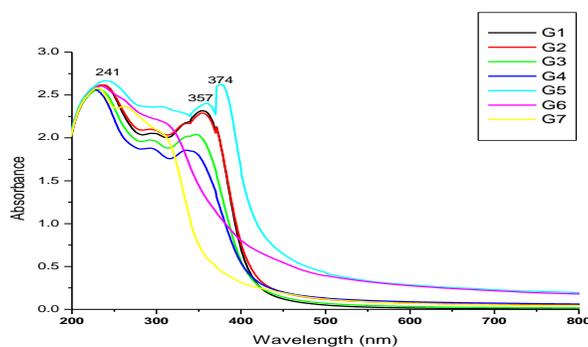
Table 2:-

UPF Range	Protection Category	UVBE _{eryt} transmittance (%)
< 15	Insufficient protection	> 6.7
15-24	Good Protection	6.7-4.2
25-39	Very Good protection	4.1-2.6
40-50, 50+	Excellent Protection	≤ 2.5

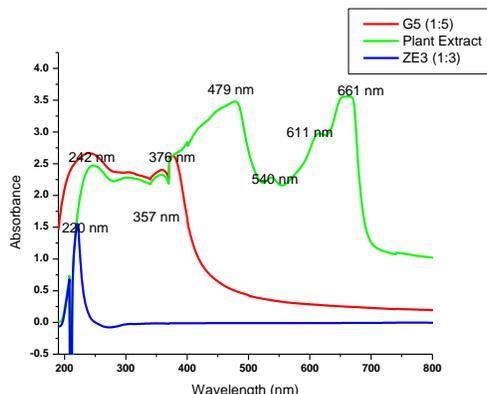
Results and Discussion:-

Ultraviolet Visible Spectroscopy:-

Confirmation and monitoring of the reduction of zinc ions to zinc oxide nanoparticles was UV-Vis spectrum in the range of 200-1000 nm. The spectrum showed distinct peaks of UV absorption are depends on several factors such as band gap, oxygen deficiency, size and structure of the NPs, surface roughness and impurities. [36].



(a)



(b)

Fig 5:- UV absorption shown (a) GM different concentration (b) GM G5 1:5, CM and Pure *Annona muricata* (L) ethanol extract,

UV absorption spectrum peaks shown fig 5 (a) that G5 demonstrate the high UV absorption peak 658nm, 438nm, 358nm, 246nm than other peaks. The Concentration of 1:5 samples G5 is particular are followed by the further synthesis and application process.

Fig 5 (b) shown that CM show the high UV absorption peak 220nm peak, 250nm, 300nm, has a bend. The Concentration of 1:3 samples ZE3 is particular the further synthesis and application process. The peak appearance in the absorption curves positioned the plant extract of *Annona muricata* (L) had absorbance band at 246nm, 299nm, 355nm, 438nm and 654-660 bend. The UV sunscreen function of the epidermis is mainly fulfilled by phenolic compounds, Flavonoids and hydroxycinnamic acids, which all have absorption maxima in the UV part of the spectrum. The intense absorption band around 260 nm with a weaker band above 300nm (is flavones, flavanones) 260-300nm around flavones or 360 nm flavonols. [37] There were identified the maxima wavelengths specific to polyphenols at 280-330 nm, to Flavonoids and quinines obtained by polyphenols, oxidation at 390-420 nm and chlorophylls at 600-660 nm. According to the UV-Vis spectra of each plant can observe the absorption peaks in the region 400-412 nm and 600-660 nm region, 220-280nm and 330-420 nm corresponding to phenolic acids and their derivatives (flavones, flavonols, phenylpropenes and quinones), richest in phenolic derivatives 289 nm and Flavonoids 330 nm. [38] **GM G5 1:5** at 376 nm, Absorption 255 nm for zinc nano crystals embedded into SiO₂ matrix, 358 nm corresponds to peak of ZnO shell layer [39] The UV absorption spectrum shows an absorption band at 355 nm due to ZnO nanoparticles. [40] ZnO nanopowder is exhibits a strong absorption band at about 355 nm. [41] The absorption of UV range up to 361.75 nm and almost all the visible spectrum radiations are transmitted by the ZnO nanoparticles. [42] **CM** shown at 220 nm, The UV peak at 230 nm is due to the transition of inner shell electron to the conduction, which is almost reported in the case of UV-visible absorption of all metal and metal oxide nanoparticles. [39] Absorption peak found at about 258 nm due to the ZnO nanoparticle which lie much below the band gap wavelength of 358 nm. [43] The UV visible absorption spectroscopy of ZnO nanoparticles in ethanol solvent shows an excitonic absorption peak at about 214 nm, which lies much below the band gap wavelength of 388 nm of bulk ZnO. [44,45]

XRD:-

The XRD pattern of ZnO nanoparticles is illustrated in Fig 6 (a) illustrated UV absorber GM G5 1:5 peaks located at $2\theta = 33.26^\circ, 58.78^\circ$ are associated well agreed with the JCPDS 21-1486, the powder pattern as cubi structure and the crystallite size of nanopowder around 86 nm.

(b) UV absorber CM the diffraction peak (JCPDS 89-1397) located at ZnO at $2\theta = 31.93^\circ, 34.53^\circ, 36.32^\circ, 47.82^\circ, 56.62^\circ, 62.88^\circ, \text{ and } 68.26^\circ$ are associated with (100), (022), (101), (102), (103) and (112) plains. All the reflections can be assigned to the standard powder pattern for the pure hexagonal phase structure of ZnO with lattice constants $a=3.2516 \text{ \AA}, c=5.2000 \text{ \AA}$. The particle size attained material was determined by using light scattering, the results reveals that particle sizes between 50 nm were obtained. ZnO NPs sharp peaks appearing about 2θ of $36.25^\circ, 31.7^\circ, 34.4^\circ, 47.62^\circ, 56.6^\circ, 62.8^\circ, 67.9^\circ$ were assigned to (111,100,002,102,110,103,112 and 102) plain value for JCPDS

card no 36-1451. [46] ZnO NPs (JCPDS 36-1451) $2\theta = 31.41^\circ, 34.31^\circ, 36.92^\circ, 47.79^\circ, 56.54^\circ, 62.81^\circ, 728.91^\circ$ and 77.17° these location of the characteristic Bragg reflection were indexed (111,002,311,222,400,331,420 and 422) planes of ZnO wurtzite structure. [47]

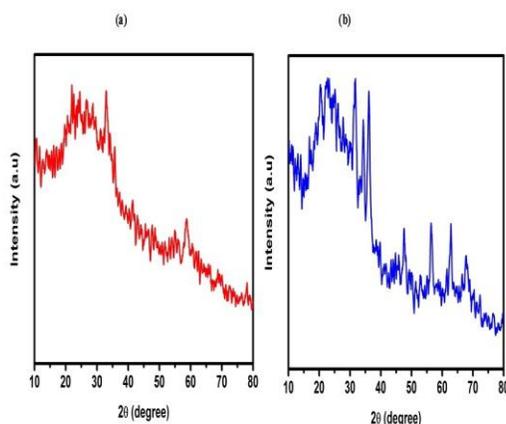


Fig 6:- X-ray diffraction (a) GM G5 1:5 and (b) CM

SEM:-

Fig 7 (a), (b) imaged by GM G5 1:5 dogged the particles size and external morphology of the ZnO NPs. It shown cubi structure composed of quite a lot of individual small nanoparticle. (c), (d) the morphology of CM is represents that the SEM images confirm the morphology of ZnO NPs show nano flake like structure. As the growth progresses nanoparticles aggregate to form a variety of morphology such as cubes, spheres, triangle, hexagons, pentagons, rods and wires. [48].

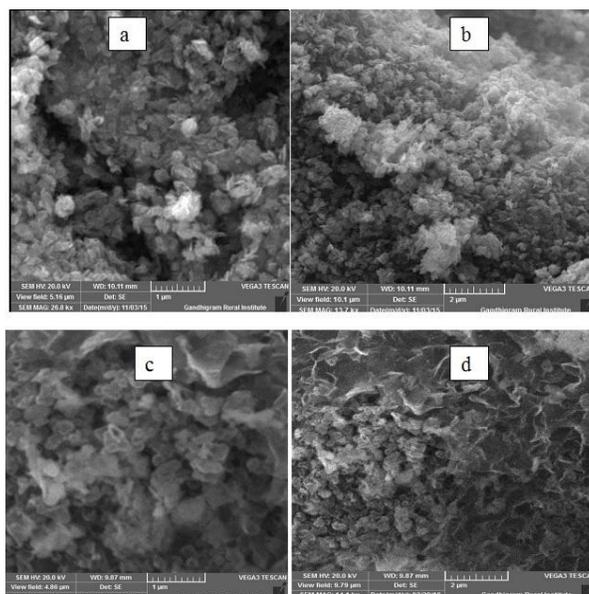


Fig 7:- shows Scanning Electron Microscopy - GM G5 1:5 (a) $1\mu\text{m}$, (b) $2\mu\text{m}$ of and CM (c) $1\mu\text{m}$, (d) $2\mu\text{m}$

EDAX:-

EDAX result further confirm the structural element analysis ZnO NPs. Fig 8 (a) GM G5 1:5 the spectrum peaks of zinc and oxygen element 63.85 and 26.15 % the developed a green and chemical route of nanoparticles using *Annona Muricata* (L) is being eco-friendly and can be an effective alternative for the large scale synthesis of ZnO NPs. The spectra also indicate like (C, K, Al, P) P.E. Ochieng *et al* 2015 [47] reported silicon, chlorine and potassium the impurities (Cl, K and Si) could be of biological origin present in the plant extract. The presences of copper and carbon elements were due to carbon coated copper adhesive used as sample holder.

Fig (b) CM indicates that are composed of zinc and oxygen of 72.89 and 24.91% proves ZnO NPs is essentially free from impurities.

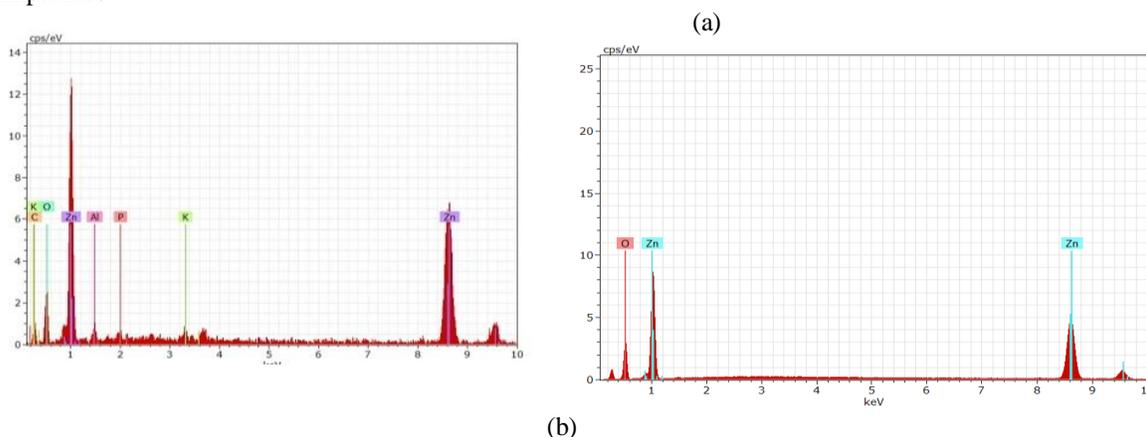
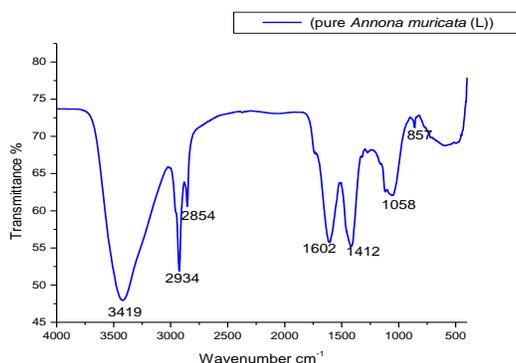


Fig 8:- EDAX (a) GM G5 1:5 and (b) CM ZE3 1:3

FTIR:-

FTIR measurements were carried out in order to determine identify for the capping and efficient stabilization of the metal nanoparticles synthesized by *Annona muricata* (L) leaf extract. The IR spectrum of ZnO nanoparticles band shown Fig 9 (a) shown pure *Annona muricata* (L ethanol extract) 3419 cm^{-1} functional group of Alcohol O-H, H bond strong intensity, 2934 cm^{-1} , 2854 cm^{-1} Alkane C-H stretching, 1602 cm^{-1} C-H and C=O stretch in polyphenol respectively. 1412 cm^{-1} Aromatic C=C multiple band, 1058 cm^{-1} Alkyl halide C-F stretch strong, 857 cm^{-1} P-subst.Benzenes.

Fig 9 (b) shown GM G5 1:5 the pattern of absorption at 462 cm^{-1} and 473 cm^{-1} at CM the band could be attributed to the metal oxygen the characteristic stretching vibration of ZnO nanoparticles. Absorption at 450-540 cm^{-1} identifies the presence of ZnO nanoparticles. [11] Band 836 cm^{-1} aromatic C-H out of plane bending is a typical nano substituted benzene ring 1,2,3, tri-substituted benzene ring and 1,4 di-substitute benzene ring. The frequencies of α -D- glucose in the region at 836 and 902 cm^{-1} . [49,50] Medium absorption in the region 1431 cm^{-1} often implies an aromatic ring. This band is absorbed CO_2 spectrum may be due to zinc carbonate, while the absence of bands in this region in either of the CO spectra indicate that in these cases no carbonate was formed [51] The band at 2360 cm^{-1} is due to CO_2 [52] for the soluble CO_2 (g) in the narrowing of band within increase in line intensity to change of structural features of the materials, this may be due to a change in crystalline and/or distortion of symmetry caused by the insertion of various foreign impurities with progressive heating temperature. [53] Broad IR bands at 3430 cm^{-1} can be attributing to the characteristic functional group alcohol O-H stretching vibration. [54] 3410-3460 cm^{-1} O (2) H--O(6) intermolecular H-bonds. [55,56] .



(a)

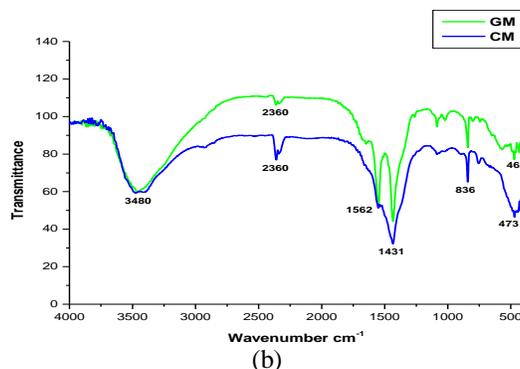


Fig 9:- FTIR (a) Pure *Annona muricata* (L) ethanol extract (b) GM G5 1:5 and CM

SEM ZnO NPs Fabric:-

Fig 10 characterize the SEM Micrographs 10 μm magnifications were represent the GM G5 1:5 and CM ZnO NPs were well diffuse on the surface in both the side of the fabric, while aggregated nanoparticles are noticeable.

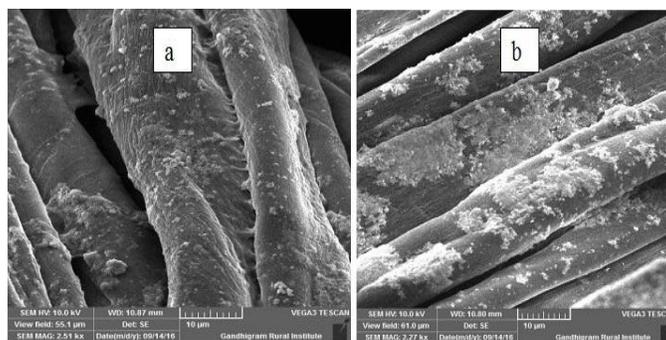


Fig 11:- (a) GM G5 1:5 (b) CM, UV absorber (ZnO NPs) coated fabric

UPF Analysis:-

Zinc oxide nanoparticles are very special among the nanomaterials due to their organometallic properties. [57] Nadanathangam vigneshwaran *et al* 2006 [58] reported the UPF factor for the control cotton fabric is calculated to be 1.54, 2.48 and 3.71 for bulk ZnO NPs coated cotton fabric, respectively two half fold increases in UPF in comparison with the control fabric. The UPF values and the per cent of UV transmission for UV-A and UV-B ranges were represent in Table 3.

Table 3:- Ultraviolet Protection Factor

Samples Code	Mean T (UVA)%	T	Mean T (UVB) %	Mean UPF	UV Protection
Gf	3.97	4.27	24.37	24.37	Good protection
Bf	5.53	6.67	12.30	12.30	Insufficient protection
BfGM _{UVAb}	4.43	3.14	30.26	30.26	Very Good protection
BfCM _{UVAb}	3.74	4.28	24.58	24.58	Good protection

Abbreviation:-

Gf - Grey fabric, Bf- Bleached fabric, BfGM_{UVAb} - Bleached Green Method UV absorber, BfCM_{UVAb} - Bleached fabric Chemical Method UV absorber.

Table 3 shows that the Cotton bleached fabric (Bf) has insufficient protection significantly lower than the standard value. It's classifying the clothing need UV-shield, These UV absorber (ZnO Nps) GM G5 1:5 and CM coated BfGM_{UVAb}, BfCM_{UVAb} both are give good protection against UV radiation, BfGM_{UVAb} give very good protection compared to BfCM_{UVAb}. Grey cotton fabric (Gf) has starch added for the warp yarns before weaving process, thus reason behind that it gives good protection. Natural plant extract been considered as potential sunscreen resource because of their ultraviolet rays absorption characteristic in the UVA, UVB region and their antioxidant activity.

There is strong evidence of DNA-damaging ultraviolet rays induces the accumulation of UV light absorbing flavonoids and other phenolics in dermal tissue of the plant body which are having excellent antioxidant and photo protective properties. [59,60] In this green method increased the concentration of *Annona muricata* (L) plant extract, it has high in flavonoid and other phenolic compounds, these reason behind GMG5 1:5 enhanced the UV absorption properties than CM UV absorber. (Fig 11).

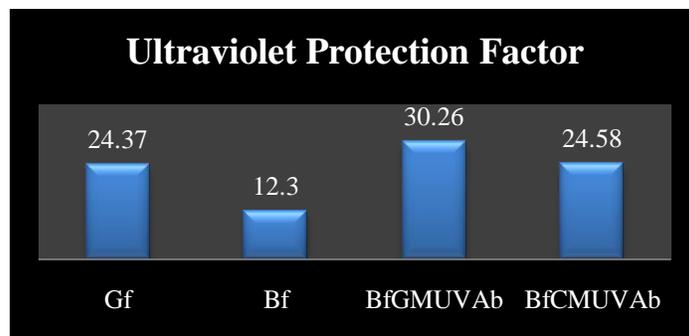


Fig 11:- Ultraviolet Protection Factor Treated/Un treated fabrics

The interesting aspect of the nanoparticles treatment is their wash fastness. The treated fabric was tested after 25 washes again and no significant change in its sunscreen activity was observed. [34] Wash fastness can be further improved with the formation of covalent bonding between nanoparticles and the fabric surface. In these cases the excellent properties are still remained after about 55 home laundering. [61]

Conclusion:-

ZnO is bio safe, bio compatible and can be used for bio medical application in coating, with these unique characteristics ZnO could be one of the most important nanomaterials in applications, Green synthesis of nanoparticles is a novel materials that are eco friendly, cost effective, stable nanoparticles with a great important for wider a application. It is a safer and best alternative conventional method. ZnO NPs embedded green synthesis potential application such as UV protection ability in textiles and sunscreen. The performance of characterization analysis GM G5 1:5 UV absorber has well, it can be concluded that the active ingredients in *Annona muricata* (L) extract and its Green method (GM) ZnO NPs are highly UV absorber due to the competitive energy absorption.

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