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Preparation of an Antimicrobial Cotton Fabric Using Synthesized Zinc Oxide Nanoparticles Stabilized by Alovera Gel

تجهيزخامه قطنيه مضاده للميكروبات باستخدام جزيئات الزنك النانوية المخلقة وتثبيتها علي الخامه باستخدام هلام الصبار

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ا لآيــة

قال تعالي :

(إِنَّ اللَّهَ لَا يَسْتَحْيِي أَن يَضْرِبَ مَثَلًا مَّا بَعُوضَةً فَمَا فَوْقَهَا ۚ فَأَمَّا الَّذِينَ آمَنُوا فَيَعْلَمُونَ أَنَّهُ الْحَقُّ مِن رَّبِّهِمْ ۖ وَأَمَّا الَّذِينَ كَفَرُوا فَيَقُولُونَ مَاذَا أَرَادَ اللَّهُ بِهَٰذَا مَثَلًا ۘ يُضِلُّ بِهِ كَثِيرًا وَيَهْدِي بِهِ كَثِيرًا ۚ وَمَا يُضِلُّ بِهِ إِلَّا الْفَاسِقِينَ)

(الآية 26 – سورة البقرة)

صدق الله العظيم

ADEDICATIONS

Firstly, I dedicated this study to my almighty god, thanks you for the guidance, strength, power of mind, protection and for giving me a healthy life, all of these offer to you.

My god

Many thanks for those how teaching me directed me and provid cared my research until finish.

My teachers

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My parents

To my sisters and friends who shared their words of advice and encouragement to finish this study

My sisters

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المستخلص

في الآونة الأخيرة ، اصبح اختيار المنتجات النسيجية من الناحية الجمالية عوضاً عن الناحية الوظيفية في ازدياد مطرد يوماً بعد يوم من قبل العملاء ، ولهذا السبب تم اختراع و تطوير تقنيات حديثة في المجالات المختلفة من انتاج المنسوجات التقنية وذلك لتحقيق رضا العملاء ، وانتاج منتج ذو أداء وظيفي جيد . وتعتبر تقنية النانو من أهم التقنيات في مجال تجهيز المنسوجات ويتم استخدامها بمعدل عالي في هذا المجال ويتم من خلالها انتاج انواع مختلفة من المنسوجات التقنية العالية الأداء والتي تستخدم في مجالات حيوية بالغة الأهمية . وتَجهز المنسوجات بعدة طرق منها : تغطية النسيج بجزيئات في حجم النانو ، أو تصنيع شعيرات وخيوط في حجم النانو ومن ثم تُحَول الى منسوجات بمواصفات وخواص تُمَكن من استخدامها في النسيج الذكي ، وتَنتَج ملابس لها خواص الحماية من الميكروبات ، وصناعة سترات واقِية من الرصاص وملابس مقاومة للابتلال ، وصناعة ملابس ضد الاتساخ وغيرها من المنسوجات الذكية.وفي هذه الدراسة تم تسليط الضوء على تقنية النانو واستخدامها في تجهيز المنسوجات ، وتم تجهيز قماش قطني باستخدام حمض الستريك والالوفيرا مع الزنك ، والتي عملت على ربط جزيئات النانو بالخامة القطنية المستخدمة لتوفير النشاط المضاد للميكروبات . طبيعة تشكل السطح والتركيب الكيميائي تم وصفه عن طريق المسح المجهري الالكتروني ، وانكسار الالكترون ، ومحول فورييه للاشعة تحت الحمراء.خاصية مقاومة الميكروبات تم قياسها ضد البكتريا المتكورة العنقودية . النتائج اظهرت ان جزئيات اوكسيد الزنك النانوية قد تجمعت بنجاح على سطح الألياف ، في وجود الالوفيرا وحمض الستريك كعوامل ربط ، والتي وزِعت بشكل متجانس وغير متجانس على سطح القماش . اختبارات مقاومة البكتريا اظهرت الخامة المغطاه بجزيئات الزنك والالوفيرا وحمض الستريك كعوامل ربط ، بانها تمتلك نشاط جيد مكافح للجراثيم والبكتريا العنقودية. الخصائص الفزيائية كالانكماش والرجوعية والاحتكاك وقوه الشد ، تم فحصها والتحقق منها باستخدام اجهزة شيرلي والكروكميتر . و اظهرت النتائج تحسناً مممتازاً في خصائص القماش.

ABSTRACT

Nano-finishing is one of the most promising candidate, which is increasing significantly now a day in textile fields, which are ranging from the protective finishing such as; smart textiles, Hygiene textiles, antiballistic, bulletproof vest. To functionalized finishing such as; water repellent, wrinkle resistance clothing, and self-cleaning. However, unfortunately through laundry, exposure to sunlight, or mechanical abrasion, textiles generally lose between 5% and 20% of their weight during their use phase. Therefore, it can be assumed that nanomaterials integrated into, functionalized, or applied onto, surface of textiles will enter to the environment when it release from textile surface during washing or mechanical abrasion. Which makes the nano-finishing of textile valueless and generates environmental hazards.

In order to enhanced the attached of nanoparticles to surface of fabric, in this work Zinc oxide (ZnO) nanoparticles were synthesized on the surface of cotton fabric via a sol-gel method, in presence of Alovera and citric acid as selective natural cross-linking agents. In order to providing antimicrobial activity and enhancing physical properties.

Surface morphology and chemical composition of finished and unfinished fabric have been examined by scanning electron microscopy (SEM), electron diffraction (EDs) and Fourier transfer infrared (FTIR). Antibacterial properties was evaluated against (Gram-negative) Ecoli and (Gram-positive) Staphylococcus aureus bacteria. The results showed that ZnO nanoparticles was successfully synthesizes on fiber surface, in the present of Alovera and Citric acid as crosslinking agent. And it's distributed homogenously and non-homogenously in the fabric surface, in case of used Alovera capping agent and non homogenies with citric acid.

Antibacterial tests showed that the ZnO-coated fabric with Alovera and Citric crosslinking possesses good bacteriostatic activity against to staphylococcus and

Ecoli. Physical properties shrinkage, crease recovery angle (CRA), abrasion and tensile test were investigated by using Shirley (CRA), crokmerter and belistone instruments, the results were showed excellent enhancement in physical properties.

Abrasion test was used to ensure that wether ZnO nanoparticles were released on surface of tested fabric or not. To confirm that, SEM and EDS used to investigate if there is any Zn elements has been detected in abrasion sample (bottom fabric surface). The result showed there is no Zn element, which means the attachment of ZnO nanoparticles to the cotton fabric was improved significantly due to the presence of crosslinking agents (Alovera, and Citric).

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CHAPTER ONE

1.1 Introduction

Nanomaterials have been defined as utilization of structure with at least one dimension of nanometer size for the construction of materials, devices or systems with novel or significantly improved properties due to their nano-size. The "nano" word comes from the Greek word "nanos" which means, "dwarf". One nanometer is one-billionth of a meter, i.e. 10^{-9} m. Nanotechnology aimed at manipulating atoms, molecules and nanosized particles in a precise and controlled manner in order to build materials with a fundamentally new organization and novel properties. The fundamentals of nanotechnology lie in the fact that properties of substances dramatically change when their particle size reduced to the nanometer range. Hence, it has been to allowing scientists, engineers, chemists, and physicians to work at the molecular and cellular levels (i.e. at nano-level) to produce important advances in the life sciences and healthcare. Currently, nanomaterials are classified into three main categories, which are: 1) nanofibers, 2) nanofibril, and 3) nanoparticles, each one of these kind of nanomaterials and varied range of applications.

Nanoparticles are particles with at least one dimension smaller than 1µm, and potentially as small as atomic and molecular length scale of 0.2 nm. Nanoparticles (of less than 100 nm) contain one million atoms or less (one nm radius can occupy approximately 25 atoms) and the majority of atoms are at the particle surface. Many properties of matter depend on the particle-size range.

The use of nanoparticle [NP] materials offers (Akbarzadeh et al., 2012) major advantages due to their unique size and physicochemical properties.

Generally, nanoparticles reduced size associated with high surface/volume ratios that increase as the nanoparticle size decreases. As the particle size decreases to some extent, many constituting atoms could be found around the surface of the particles, which makes the particles highly reactive with prominent physical properties. Due to that, nanoparticle materials have shown unique properties, with a flexibility and ability for modification. Currently, nanomaterials being applied in different areas of applications such as, biomedical, energy, water treatment, food and cosmetics and textiles. So, the innovative methods, manipulation, and control of the materials properties via means of nanomaterials and nanotechnology has been needed .

In addition, synthesis of nanoparticles having uniform shape and size via easy synthetic routes is the main issue in nanoparticle growth For the past decade, scientists have been involved in the development of new synthetic Routes enabling the precise control of the morphology and size of the nanoparticles. In general, nanoparticle have been synthesized via liquid (chemical method), solid, and gaseous media but due to several advantages over the other methods, chemical methods are the most popular methods due to their low cost, reliability, and environmentally friendly synthetic routes, and this method provides rigorous control of the size and shape of the nanoparticles. In general, nanoparticles could be synthesized via top-down or bottom-up methods as it shown in fig1.1.

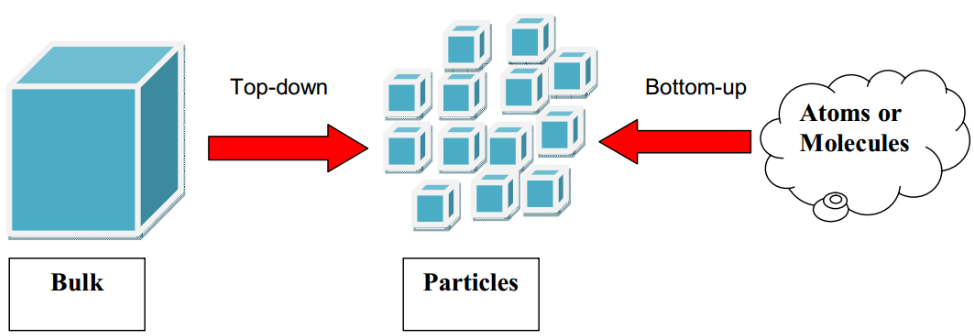


Fig (1.1): synthesized nanoparticles

Nanoparticles have a high surface-to-volume ratio are needed, but the agglomeration of small particles precipitated in the solution is the main concern in the absence of any stabilizer. In this regard, preparations of stable colloids are important for nanoparticle growth. In addition, nanoparticles were stabilized by steric repulsion between particles due to the presence of surfactant, polymer molecules, or any organic molecules bound to the surface of nanoparticles. Sometimes van der Waals repulsion (electrostatic repulsion) also plays important role in nanoparticles stabilization. With all the issues related to nanoparticle synthesis, there are various types of nanoparticles reported in the literature, e.g., metal nanoparticles, metal oxide nanoparticles, and polymer nanoparticles. Among all these, metal oxide nanoparticles stand out as one of the most versatile materials, due to their diverse properties and functionalities. Most preferentially, among different metal oxide nanoparticles, zinc oxide (ZnO) nanoparticles have their own importance due to their vast area of applications, e.g., gas sensor, chemical sensor, bio-sensor, cosmetics, storage, optical and electrical devices, window materials for displays, solar cells, and drug-delivery (Mariana, 2015).ZnO is an attractive material for short-wavelength optoelectronic applications owing to its wide band gap. As a wide band gap material, ZnO could be use in solid-state blue to ultraviolet (UV) optoelectronics, including laser developments. In addition, due to its non-centrosymmetric crystallographic phase, ZnO shows the piezoelectric property, which is highly useful for the fabrication of devices, such as electromagnetic coupled sensors and actuators (Tang et al., 2004).

Textile industry, being made a great impact to many aspects of the life and society. New technologies in material science need to apply to the traditional textile industry to develop and produce high value added products and overcome the problems associated with the conventional finishing treatments. Conventional textile finishing have been relatively long history and well established. However, with the ever - increasing demands from the consumers and the society, the conventional treatments are unable to cope with these demands since many of them alter or harden the hand, some of them have harmful effects to human and cause pollution to the environment. Last decades, application of nanoparticles in textile finishing has been used to enhance the physical properties of conventional textiles materials, these finishing includes; anti-microbial properties, water repellence, soil-resistance, anti-static, anti-infrared and flame-retardant properties, dye-ability, color fastness and strength of textile materials.

The application of inorganic nanoparticles such as, TiO2, ZnO, Ag ..etc, into textile materials aimed to produce finished fabrics with different, unique, new and multi-function properties, i.e ZnO nanoparticles can be used for antibacterial and UV-blocking properties.

1.2 Problem statement: -

The future for textile applications using nanotechnology is exploding due to various end uses like protective textiles for soldiers, medical textiles and smart textiles. Recently, ZnO nanoparticles have been applied, intensively in textiles finishing as UV-blocked, self-cleaning, and antimicrobial agents. In general, textiles lose between 5% and 20% of their weight during their use phase(Becheri et al., 2008b), e.g. through laundry, exposure to sunlight, or mechanical abrasion. Therefore, it can be assumed that nanomaterials integrated into, functionalized, or applied onto, surface of textiles will enter to the environment when it release from textile surface during washing or mechanical abrasion. Studies of the behavior of nanoparticles in textiles showed that the released of nanoparticles from textiles during laundry, abrasion and thermal treatment are depending on the way they are integrated. Release during laundry (Becheri et al., 2008b) varies from zero to almost 100%. This make the finishing of textile valueless and generates environmental hazards. There are some of the nanoparticles effects on living systems of human that may be related with the size of the dimensions of nanoparticles. For example, nanoparticles with few nanometer dimensions less than 1000 nm, which it may reach inside biomolecules and cross the cell membranes (human blood cell, 7000 nm, human skin cell in 25-40 micrometers)(Devreese, 2007) . Inhaled may reach liver, heart or blood cells. In case, these nanoparticles exposed to sunlight may catalyze oxidative damage to DNA and cell, which it leads to cancer infection. Therefore, nanoparticles assumed to be stably embedded to maintain the product quality and functionality of the textiles. This also prevents the release of nanomaterials with potential hazards to human health and environment. Hence, the possibly the highest risks of nanotechnology to humans and environment can be related with the free nanoparticles. Therefore, to overcome the immigration of nanoparticles from the surface of textile to surrounding environment, some of organic molecules and natural extract from plant have been proposed to use as fabric surface modifier, which act as cross-linker to enhance the adhesion between the textile surface and nanoparticles, due to their chemical structure.

In this work, the extracted material from alovera and citric acid are suggested to be use as crosslinker to modify the fabric surface to enhance the adhesion between fabric and nanoparticles to improve the distribution of nanoparticles on the fabric's surface and to eliminate the aggregation of nanoparticles as well.

1.3 Plan and experiment design:

In this work, alovera and Citric acid were used as fabric surface modifier (crosslinker) to enhance the attachment of ZnO nanoparticles on the surface of fabric, and to serve as a capping agent/stabilizer to prevent the aggregation of ZnO nanoparticles during the synthesis. This will leads to supporting the durability of ZnO on the surface of cotton fabric during the use, and enhancement the anti-microbial properties of the cotton fabric as well.

CHAPTER TWO

Literature review

2.1 Introduction

The term of “Nano” comes from the Greek word “Nanos” meaning “dwarf” and it used in the measuring system as a prefix to denote one billionth. A particle with a diameter of one nanometer is therefore 1 billionth of a meter in size (10^{-9} m = 10^{-6} mm). The history of nanotechnology is generally understood to have begun in December 1959 when physicist Richard Feynman gave a speech, “There's Plenty of Room at the Bottom”, at an American Physical Society meeting at the California Institute of Technology in which he identified the potential of nanotechnology. Feynman said it should be possible machines small enough to manufacture objects with atomic precision. In 1974, Norio Taniguchi first used the word “nanotechnology, in regard to an ion sputter machine, to refer to “production technology to get the extra-high accuracy and ultra-fine dimensions, i.e. the preciseness and fineness on the order of one nanometer.” In the 1980s, Eric Drexler authored the landmark book on nanotechnology, “Engines of Creation”, in which the concept of molecular manufacturing was introduced to the public at largely. By the 1990s, nanotechnology was advancing rapidly(Akbarzadeh et al., 2012) .Nowadays, there are many scientists whom thinking that the next Industrial Revolution is coming soon because of nanotechnology. which will radically transform the world, nature, and the people life. (Mariana, 2015)

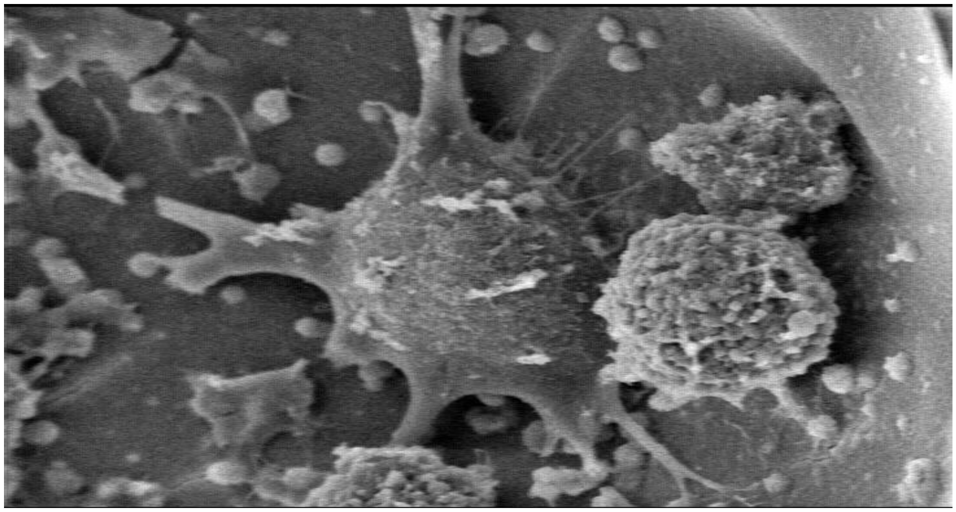


Fig (2.1): Nanoparticles shape

2.2 Nanomaterials:-

Nanomaterial is defined as the material with one or more external dimensions, or an internal structure, which could exhibit novel characteristics compared to the same materials without nanoscale features. There are several definitions of nanomaterials and the products of nanotechnology, often these definitions have generated for specific purposes. Nanotechnology is the term given to those areas of science and engineering where phenomena that take place at dimensions in the nanometer scale are utilized in the design, characterization, production, and applications of materials, structures, devices and systems (Mariana, 2015).

2.2.1 Classification of nanomaterials:-

Nanomaterials are characterized by free nanoparticles of various nano-dimensions 1D,2D and 3D in classifying nano sheets such as graphene, 2D needles or filament ,3D particles including aggregates.(Murr, 2015)

a. One-dimensional nanostructure (ID)

The smallest possible crystalline wires with cross-section as small as a single atom can be engineered in cylindrical confinement. Carbon nanotubes, a natural semi-one dimension nanostructure, can be used as a template for synthesis. Confinement provides mechanical stabilization and prevents linear atomic chains from disintegration; other structures of 1D nanowires are predicted to be mechanically stable even upon isolation from the templates.

b. Two-dimensional nanostructure (2D)

2D materials are crystalline materials consisting of a two-dimensional single layer of atoms. The most important representative graphene was discovered in 2004(Tung et al., 2009). Box-shaped graphene (BSG) nano structure is an example of 3D nanomaterial (Lapshin, 2016).BSG nanostructure has appeared after mechanical cleavage of pyrolytic graphite. This nanostructure is a multilayer system of parallel hollow nano-channels located along the surface and having quadrangular cross-section. The thickness of the channel walls is approximately equal to one nm. The typical width of channel facets makes about 25 nm. Thin films with nanoscale thicknesses are considered nanostructures, but are sometimes not considered nanomaterials because they do not exist separately from the substrate.(Lojkowski et al., 2006, Klaessig et al., 2011)

c- Bulk nanostructured materials

Some bulk materials contain features on the nanoscale, including nanocomposites, nanocrystal line materials, nanostructured films, and Nano textured surfaces .(Klaessig et al., 2011, Lojkowski et al., 2006)

Currently, nanomaterials are classified into three main categories, which are, 1) nanofibers, 2) nanoparticles. each one of these kind of nanomaterials has varied range of applications

1- Nanofibers:-

It knows as the fibers with diameters in the nanometer range. Nanofibers can be generated from different polymers and hence have different physical properties and application potentials. The diameters of nanofibers depend on the type of polymer used and the method of production.(Reneker and Chun, 1996) All polymer nanofibers are unique for their large surface area-to-volume ratio, high porosity, appreciable mechanical strength, and flexibility in functionalization compared to their microfiber counterparts.(Khajavi et al., 2016, Vasita and Katti, 2006, Li and Xia, 2004) ,There are many different methods to make nanofibers, including drawing, electrospinning, self-assembly, template synthesis, and thermal-induced phase separation. Electrospinning is the most commonly used method to generate nanofibers because of the straightforward setup, the ability to mass-produce continuous nanofibers from various polymers, and the capability to generate ultrathin fibers with controllable diameters, compositions, and orientations.(Li and Xia, 2004). This flexibility allows for controlling the shape and arrangement of the fibers so that different structures (*i.e.* hollow, flat and ribbon shaped) can be fabricated depending on intended application purposes. Nanofibers have many possible technological and commercial applications. It has been used in tissue engineering, drug delivery, cancer diagnosis, lithium-air battery, optical sensors and air filtration. In nanofibers, Polymer chains are connected via covalent bonds.(Teraoka and Teraoka, 2002, Dey and Pridham, 1972), weather it is natural polymers such as collagen, cellulose, silk fibroin, keratin, gelatin and polysaccharides such as chitosan and alginate.(Vasita and Katti, 2006). Or synthetic polymers such as poly lactic acid (PLA), polycaprolactone (PCL), polyurethane

(PU), poly(lactic-co-glycolic acid) (PLGA), poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), and poly(ethylene-co-vinylacetate) (PEVA).(Khajavi et al., 2016, Vasita and Katti, 2006).

2- Organic or Inorganic Nanoparticles:-

Nanoparticles are particles between 1 and 100 nanometers (nm) in size. Interfacial layer is an integral part of nanoscale matter, fundamentally affecting all of its properties. The interfacial layer typically consists of ions, inorganic and organic molecules. Organic molecules coating inorganic nanoparticles are known as stabilizers, capping and surface ligands, or passivating agents.(Li and Xia, 2004) In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties. Particles are further classified according to diameter.

2.3 Nanotechnology:-

Nanotechnology*:* the design, characterization, production and application of structures, devices and systems by controlling shape and size at the nanoscale. Nanotechnology is science, engineering and technology conducted at the nanoscale which is about 1 to 100 nanometers. Nanotechnology is the study application of extremely small things and can be used across all the other science fields, such as chemistry, biology, physics, materials science, and engineering (Lieber, 2003). It is the understanding and control of matter at dimensions between approximately 1 and 100 nanometers, where unique phenomena enable novel applications. Encompassing nanoscale science, engineering, and technology, nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale. A nanometer is one-billionth of a meter. A sheet of paper is about 100,000 nanometers thick; a single gold atom is about a third of a nanometer in diameter. Dimensions between approximately 1 - 100 nanometers are known as the nanoscale. Scientists have not unanimously settled on a precise definition of nanomaterials, but agree that they are partially characterized by their tiny size, measured in nanometers. A nanometer is one millionth of a millimeter - approximately 100,000 times smaller than the diameter of a human hair. Materials engineered to such a small scale are often referred to as engineered nanomaterials, which can take on unique optical, magnetic, electrical, and other properties. These emergent properties have the potential for great impacts in electronics, medicine, and other fields.(Mariana, 2015)

2.4 Nanoparticles: -

Nanoparticles are a single part of nanomaterials that are defined as 1–100 nm in diameter. According to the British Standards Institution (BSI 2005).

Nanoparticles can be classified as organic and inorganic particles, which could be applied alone or in combination with each other as nanocomposites to provide multifunctional properties. Inorganic nanoparticles are further grouped into metals such as silver, gold, copper, nickel, and metal oxides, including titanium dioxide, zinc oxide, iron oxide, and copper oxide. Recently, semi-metals also have been considered.

On the other hand, there are organic nanoparticles as solid particles composed of organic compounds (mainly monomeric or polymeric) ranging in diameter from 10nm to 1µm such as nanochitosan, nanocellulose, nanowool, nanosilk, and denderimers. There are also some researches on the co-application of organic and inorganic nanoparticles on textile substrates such as Ag/chitosan nanocomposite. Various nanoparticles materials used in textile nanofinishing have been summarized in (fig 2-2).

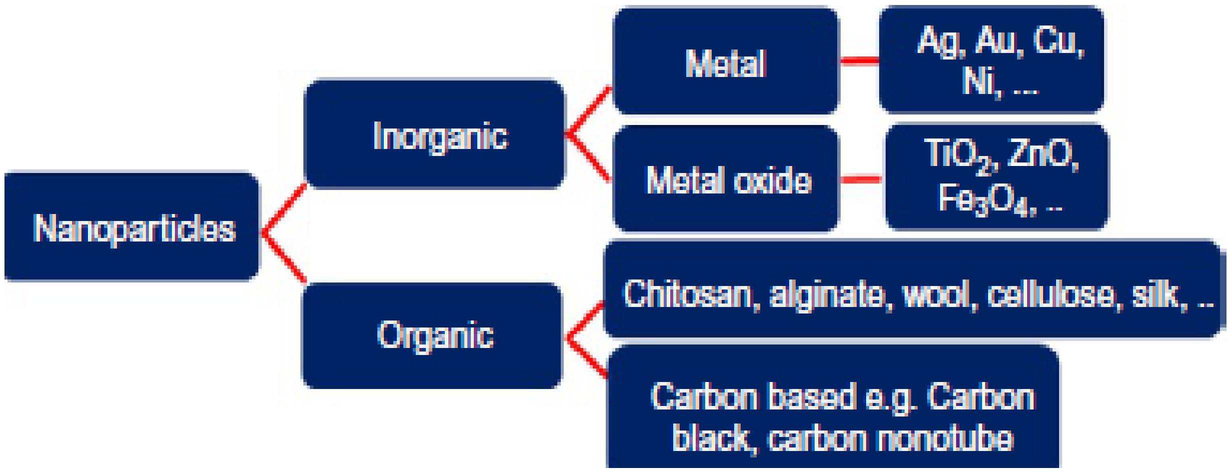


Fig (2.2): Classification of Nanoparticles

2.4.1 Properties of Nanoparticles: -

The unique properties and avail of nanoparticles also arise from a variety of properties, like the similar size of nanoparticles and biomolecules such as proteins and polynucleic acids (Akbarzadeh et al., 2012). In addition nanoparticles can be architect with a wide range of metals and semiconductor core materials, that give useful properties such as fluorescence and magnetic behavior(Becheri et al., 2008a) .Moreover, unlike their bulk counterparts, nanoparticles have reduced size associated with high surface or volume ratios that increase as the nanoparticle size decreases. As the particle size decreases to some extent, a huge number of constituting atoms can be found around the surface of the particles, which makes the particles highly reactive with salient physical properties. Nanoparticles of some materials show unique material properties, hence, fashioned, manipulation and control of the material properties *via* mechanistic means is needed.

Table (2.1): Characteristics of in Organic Nanomaterials

In organic nano structured- materials	characteristic
Titanium dioxide and inorganic or in organic modified titanium dioxide	Anti -bacterial, photo-catalyst, self- cleaning, UV protecting, Water and air purifier, dye degradation , gas sensor , solar cell, hydrophilic ,super hydrophobic, co- catalyst for cotton cross linking , photo stabilizing wool.
Silver	Anti- microbial , disinfectant, electrical conductive , UV- protection ,Anti-fungal.
Zinc oxide	Anti -bacterial, UV-photo blocking, super hydrophobic , photo-catalyst.
Copper	Anti -bacterial, UV- protection, electrical conductive
Clay	Anti- bacterial , flame retardant , UV- absorber
Gold	Anti-bacterial , Anti- fungal , electrical conductive
Gallium	Anti- bacterial
Carbon nanotubes(CNT)	Anti-microbial, electrical conductive, fire retardant, Anti-static, chemical absorber.

2.4.2 Synthesis of Nanoparticles: -

Nanoparticle synthesis can be possible *via* liquid (chemical method), solid, and gaseous media (Polarz et al., 2007), chemical method is the most popular due to their environmental friendly, reliability and low cost. This method provides rigidly control of the shape and size of the nanoparticles. In general, nanoparticles with high surface-to-volume ratio gives good properties, but the agglomeration of small particles in the solution is the main concern in the absence of any stabilizer. To avoided agglomeration, preparations of stable colloids are important for nanoparticle growth. In addition, nanoparticles could stabilized by repulsion between particles due to the presence of surfactant, sometimes van der Waals repulsion (electrostatic repulsion) also plays important role in nanoparticles stabilization.

2.4.3 Application of nanomaterials and nanoparticles:-

In recent time, nanoparticles have been a common material for the development of new cutting-edge applications in, energy storage, optics, sensing ,data storage , transmission, environmental protection, cosmetics, biology, and medicine due to their important electrical ,optical , and magnetic properties.(Li et al., 2008)

2.5 Nanotechnology in Textiles:-

2.5.1 Introduction:-

Due to the rabidly progress of nanotechnology in the manufacturing of fibers/yarns (nano spinning) including the development of fabric finishes, the applications and disciplines of nano technology are pervasive in the area of textiles for the last few decades. fabric finishes is greatly contributed to the advancement in the area of nanotechnology. By combining the nanoparticles with the organic and inorganic compounds, the surfaces of the fabrics modified to hydrophobic, abrasion resistant, ultraviolet (UV), electromagnetic and infrared protection finishes can be significantly modified e.g Titanium-dioxide (TiO_2) nanoparticles have been utilized for the UV protection. The usage of nanoengineered cross-linking agents during finishing process enhances the wrinkle resistance of cotton fabrics(Sawhney et al., 2008) . The recent developed micro encapsulation technique is being used in textile industry for flame or fire retardant agents and bactericidal (Montazer and Harifi, 2018), e.g. Microcapsules of silver nano particles have been used for providing anti-microbial effects and for odor control. Nanoscale fibrous materials, Nanotextiles**,** are materials that can be functionalized with a vast array of novel properties, including antibiotic activity, self-cleaning and the ability to increase reaction rates by providing large surface areas to potential reactants. These materials are used not only as cloth fabric but also as filter materials, wound-healing gauzes and antibacterial food packaging agents in food industry.(Gao et al., 2005) Application of nanoparticles in textile industry helps in developing existing performance and functions of textile materials and making intelligent textiles with new characteristics and properties and open new field of applications. However there are various nanofinishes have been applied in textile field, such as;

1-UV Protection Finishing

Nano clay flakes, composed of hydrous Alumnio oscillates, and metal oxides such as, Titanium dioxide, and zinc oxide have been used for UV protection in textile finishing. This refer to their heat, chemical, and electrical resistance properties. The presence of these nanoparticles in fabric, acting as UV absorbing which leads to block harmful rays (Ultra-violet rays) from skin.(Wong et al., 2006)

2-Anti-bacterial finishing

To keep clothes and fabrics clean from fungal and bacterial, Titanium dioxide, zinc oxide, and silver particles can help to achieve these properties in such textile finishing. Silver is the most common using as anti –bacterial due to the large surface area, and when they come in contact with bacteria or fungi inhibit cell growth in addition silver encapsulated dressing health products are used for healing wounds, burns, and scalds(Melaiye et al., 2005) .

3-Hydrophobic finishing

This finishing, could be done by locating hydrocarbons Nano-whiskers, to the surface of cotton fabric to create a peach fuzz effect without lowering the strength. Thus, water remains on the top of the whiskers and above the surface of the fabric.

However, water can still pass through the fabric if pressure is applied. The performance is permanent while maintaining breath ability.(Wong et al., 2006)

4- Self-cleaning finishing

The cotton fabric made up from huge numbers of fibers with cylindrical structure, nanofinishing applied on this structure. At nano scale cotton fibers by using nano technique look like tree trunks, these tree trunks are covered in a fuzz of minute whiskers, which creates a cushion of air around the fiber. When the water come from fabric it is grains on the points of the whiskers, the grains compress the air in the cavities between the whiskers creating extra floatable. In technical terms, the fabric has become super- hydrophobic .in addition few whiskers points contact for dirt , the dirt adheres to the water far better than it adheres to the textile surface and is carried off with the water as it beads up and rolls off the surface of the fabric. Thus, the concept of "Soil-cleaning(Yadav et al., 2006) .

5- Finishing for wrinkle free

Nano-Tex has launched a new nanotechnology-based wrinkle-free treatment(Harifi and Montazer, 2012) in order to offer an improved performance while preserving fabric strength and integrity – providing an alternative to harsh traditional processes. Chemicals and processing methods reduce a fabric's tear and tensile strength. This means there are certain fabrics and garments that are Wrinkle-free textiles are popular and convenient for time-pressed consumers, but traditional not candidates for wrinkle-free technology, such as lightweight fabrics or slim fitting garments. Sometimes fabrics also need to be over-engineered or "beefed up" in order to withstand the fiber degradation caused by traditional wrinkle-free solutions.The nano-scale molecular structure in Nano-Tex's new Fortify DP technology penetrates more deeply in the fiber to improve wrinkle-free performance. Additionally, it uses a longer and more flexible cross-linking chain which reduces fiber stress under tension, thus reducing the significant strength loss associated with traditional wrinkle-free chemistry.

2.6 Zinc Oxide Nanoparticles (ZnO):

Nanoparticles of ZnO defined as single part of zinc oxide with diameter 1–100 nm.

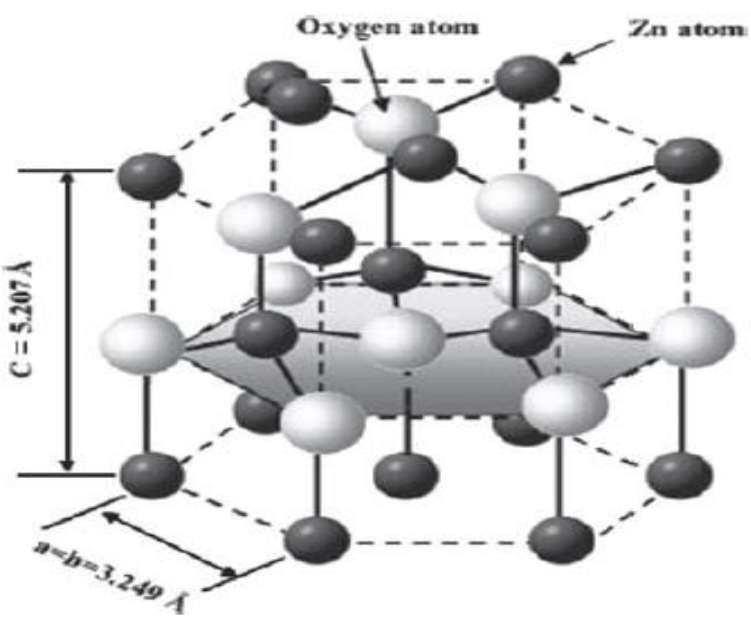


Fig (2.3): hexagonal structure of ZnO(Vaseem et al., 2010)

2.6.1 Properties of ZnO nanoparticles:-

Zinc oxide nano particles is widely used in different disciplines because of its super photocatalytic, optical, electrical electronic, dermatological , antibacterial properties, bio friendly absorbers of UV radiation.(Mohammad et al., 2010)

Zinc oxide due to their unique properties there is a possible wide range of applications common in textile finishing. ZnO has three key advantages. First, it is a semiconductor with a direct wide band gap of 3.37 eV and a large excitation binding energy of 60 meV. It is an important functional oxide, exhibiting excellent photo-catalytic activity. Secondly, because of its non-central symmetry figure 2.3, ZnO is piezoelectric, which is a key property in building electromechanical-coupled sensors and transducers. Finally, ZnO is bio-safe, biocompatible and can be used for biomedical applications without coating. With these three unique characteristics, ZnO could be one of the most important nanomaterials in future research and applications.

Table (2.2): ZnO physical properties

Properties	ZnO
Lattice parameters at 300K	
-a_0 (nm)	0.32495
-c_0(nm)	0.52069
-c_0/-a_0	1.602(1.633)
Density (g/cm^3)	5.606
Stable phase at 300K	Wurtzite
Melting point (C^0)	0.6 ,1-1.2
Thermal conductivity (Wcm^{-1} C^{-1})	a_0 :6.5 cm^3*10^{-6}
Linear expansion coefficient (C^0)	c_0 :3.0cm^3*10^{-6}
Static dielectric constant	8.656
Reactive index	2.008
Band gap (RT)	3.370
Band gap (4K)	3.437
Excision pending energy (Me V)	60
Electron effective mass	0.24
Electron hall mobility at 300K (cm^2 /Vs)	200
Hole effective mass	0.59
Hole hall mobility at 300K (cm^2 /Vs)	5-50

2.6.2 Synthesis of ZnO nanoparticles: -

Various synthetic methods have been employed to grow a variety of ZnO nanostructures, Due to its vast areas of application including nanoparticles, nanowires, nanorods, Nano belts, nanotubes, and other complex morphologies (Song et al., 2007, Gao et al., 2005, Hu et al., 2003, Zhang et al., 2002, Tang et al., 2004, Greene et al., 2003, Kong et al., 2004, Qian et al., 2003, Zhong and Knoll, 2005, Li et al., 2004, Polarz et al., 2007) . ZnO nanoparticles synthesized by the sol–gel method (solution method). Most of the literature for ZnO nanoparticles is based on the solution method. As the solution method presents a low cost and environmentally friendly synthetic route in addition, synthesis of ZnO nanoparticles in the solution requires a well-defined shape and size of ZnO nanoparticles. (Mohammad et al., 2010)

2.7 Application of ZnO in textile finishing:-

2.7.1 Introduction

Textile nano finishing define as creates many desirable properties in the fiber/ fabrics without a significant increase in weight, thickness or stiffness .However Textile finishing divided in two types:

1-Wet finishing

2- Dry finishing

Wet finishing, such as dying, which produce large amounts of contaminated wastewater. Dry finishing processes like lamination and coating often consume large amounts of energy, and are limited by the amount of material that can be applied to a fabric without adversely affecting its properties. Using nanofinishing over comes all the drawbacks of wet and dry finishing.

2.7.2 Review of Textile Finishing with ZnO:-

Rezwan Mahmud and Farhatun Nabi, (Rezwan Mahmud et.al, 2017) were investigated the cotton fabrics treated with bulk-ZnO or nano-ZnO showed different physical and mechanical properties. This reflects the improved properties of nano-sized particles with respect to conventional materials. The result showed that air permeability was reduced when the coating process was carried out with bulk-ZnO, while it was improved when nano-ZnO was used.

In another work done by Skathirvelu and et all (Kathirvelu et al., 2009), ZnO has shown a good protected against Uv radiation. they applied uv test on cotton fabric

,cotton /polyester fabric before and after washing and found significant improvement in the uv absorbing activity when the Zno nanoparticles was applied.

Yadav and et al (Yadav et al., 2006) were proved air permeability and uv blocking were improved when the nanoparticles with average size of 40 nm were coated on bleached cotton fabric (75%) UV blocking was recorded for the cotton fabrics treated with (2%) ZnO nanoparticles and the result shown that air permeability of the Nano-ZnO coated fabrics was significantly increased compered to control, hence the increased breathability.

Mohammad Shateri-Khalilabad et.al (Shateri-Khalilabad and Yazdanshenas, 2013, Shaban et al., 2018) studied anti-bacterial and ultraviolet protection factor (UPF) for Zinc oxide. (ZnO) nano-structures were synthesized on surface of cotton fabric according to AATCC Test Method 183-2004 and (UPF) value in the range of 280 – 400 nm. The results showed a good bacteriostatic activity against Gram-negative klebsiella pneumonia and Gram-positive staphylococcus aureus bacteria the UPF value of the ZnO-coated fabric increased significantly, which demonstrate ZnO nanoparticles have excellent ability to block the UV radiation.

Cotton fabric (honeycomb – weave) was coated with the synthesized ZnO nanoparticles with average size 50 nm was under go to anti-microbial test, this work was done by Mrs. S. Anita and et. Al, and the result showed coated fabric with 2% ZnO particles have high anti-bacterial efficiency . (Asokan et al., 2010)

In other work done by Nadanathangam, Vigneshwaran and et.al, prepared nanocomposite using ZnO with starch, applied on cotton fabric, and studied antibacterial and UV-protection functions. The result showed the nano-ZnO impregnated cotton fabrics have excellent antibacterial activity against two representative bacteria, Staphylococcus aureus (Gram positive) and Klebsiella pneumoniae (Gram negative). Also, nano-ZnO impregnation enhanced the protection of cotton fabrics against UV radiation in comparison with the untreated cotton fabrics (Vigneshwaran et al., 2006).

ZnO nanoparticles were applied to cotton and wool fabrics in order to evaluate the sun screen activity in the treated textiles through UV spectrophotometry. This work done by Alessio Becheri and et. al (Becheri et al., 2008b) The thermal behavior of the fabrics was assessed through thermos gravimetric analysis (DTGA), and the mechanical resistance was evaluated through tensile strength and elongation tests and they found performance of ZnO nanoparticles as UV-absorbers , can be effectively transferred to fabric materials through the application of ZnO nanoparticles on the surface of cotton and wool fabrics. Result indicates the ZnO nano particles can be applied as protection agent for human body against solar radiation and this decrease hazard of diseases injures.

(Hames et al., 2010) also reported that ZnO nanoparticles could be a good candidates for applications in solar cells, photo detectors, and displays(Dal et al., 2017) .

Vedat Dala and et.al(Dal et al., 2017) examined thermal comfort properties of twill weave cotton woven fabric coated zinc oxide layer. Results showed that thermal resistance of ZnO coated fabric increased approx. 36–52%, while thermal conductivity is decreased approx. 27–34% and WVP (water vapor permeability) is decreased approx. 22–28%.

Zinc oxide is widely used in different areas and it had good properties such as photocatalytic, electrical, electronic, optical, dermatological, and antibacterial properties (Becheri et al., 2008b, Gowri et al., 2016). Although of these unique properties there is some disadvantages of ZnO one of these was a big concern to scientists it is agglomerations. To overcome ZnO agglomerations many researchers using stabilizers agent chemicals and natural. Hence focused of natural stabilizers due to availability, low cost and bio friendly. Alovera and fruits acid were commonly used as stabilizer to stop aggregate of nano particles and keep it in homogenous distribution these promote homogenous innovated properties in each point on surface area, also make across linker to enhance stability, abrasion resistant and durability against wash. .

Li et.al., were investigated the durability of anti-bacterial activity of ZnO functionalized cotton fabric to sweat. They have treated cotton fabrics at a concentration of 11 g/L ZnO and padded them to 100% wet pick-up. The durability of anti-bacterial activity of the finished fabric in alkaline, acidic and inorganic salt artificial sweat solution has been evaluated. Results showed better salt and alkaline resistances than acid resistances for the treated fabrics (Li et al., 2007) . negative surface charge has been deduced for ZnO nanoparticles and illumination can increase anti-bacterial performance compared to normal conditions (Li et al., 2007).

ZnO nanoparticles embedded in polymer matrices like soluble starch are a good example of functional nanostructures with potential for applications such as UV-protection ability in textiles and sunscreens, and antibacterial finishes in medical textiles and inner wears(Vigneshwaran et al., 2006) these results were obtained by N Vigneshwaran, S Kumar, AA Kathe.

Xu and Cai (Xu and Cai, 2008) have grown ZnO nano-rod on cotton fabric samples through the dip-pad-cure process (Xu and Cai, 2008) However, the control mechanism of nano-rod growth has not been described. They have tried to cover the prepared nano-rod with a super hydrophobic agent to produce a cotton fabric with super hydrophobic properties based on the Cassie and Baxter theory(Dastjerdi and Montazer, 2010) .

Good antimicrobial activity of textiles was confirmed by Busila et al.(Shalaby, 2004) .They incorporated Ag and Ag:ZnO nanoparticles/chitosan biocomposites into the cotton and cotton/polyester structure using „pad-dry-cure" technique.

Alovera:

Aloe vera is a stemless or very short-stemmed plant growing to 60–100 cm (24–39 in) tall, spreading by offsets. The leaves are thick and fleshy, green to grey-green, with some varieties showing white flecks on their upper and lower stem surfaces (Guide, 2002).The margin of the leaf is serrated and has small white teeth. The flowers are produced in summer on a spike up to 90 cm (35 in) tall. The species has a number of synonyms: A. barbadensis Mill, Aloe indica Royle, Aloe perfoliata L. var. vera and A. vulgaris Lam. It is naturally growing in warm places of the world. Alovera is used in traditional medicine as a skin treatment, Cosmetic, the artificial fertilization, and textile finishing, (King et al., 1995). E.g. Micro encapsulation technology helps to add Alovera in the fabrics creating endless possibilities in the textile segment. Aloe Vera content is embedded into airtight and waterproof microcapsules. This type of fabric is mainly used in manufacturing inner garments, as they are next to the skin. In addition, Alovera has some additional functions like absorbing bad smell, and providing anti-bacterial features. They are used in the manufacture of under garments, stockings etc. (www.freepatentsonline.com).

Fig (2.4): Alovera plant

Alovera chemical composition

The leaf of this plant contains over 75 nutrients and 200 active compounds such as 20 minerals, 18 amino acids and 12 vitamins.(http://www.resil.com)

Table (1.1): Alovera chemical composition (Subhash et al., 2014)

Composition	Constituents	Action
Vitamins	Vitamins A, B and C	Acts as anti-oxidant and neutralizes free radicals
Enzymes	Aliilase, alkaline phosphatase, amylase, bradykinase, carboxylase, cellulase, lipase, peroxidase	Reduces inflammation and helps in metabolism
Minerals	Calcium, chromium, copper, selenium, sodium and zinc	Acts as co-enzymes that are essential for various enzymatic activities of metabolism
Sugars	Glucose, fructose and polysaccharides, a glycoprotein alprogen	Source of nutrition and anti-allergic action
Anthroquinones	Aloin and emodin	Acts as analgesics, anti-bacterial and anti-viral
Fatty acids	Plant steroids such as cholestrol, campesterol, beta-sisosterol and lupeol	Antiseptic and analgesic properties
Harmones	Auxins and gibberellins	Wound healing and have anti-inflammatory action

Alovera chemical structure

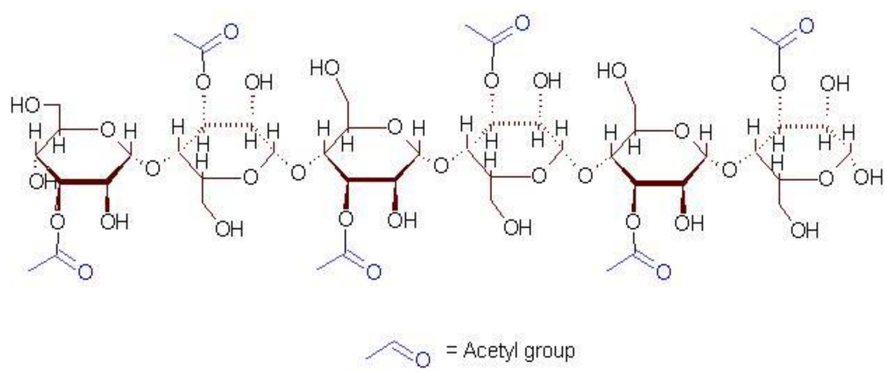


Fig (2.5): Alovera chemical structure

Alovera application:

Wound healing

When Aloe is combined with other antimicrobials, a wound heals faster than with the anti-microbial alone, possibly due to its moisturizing capability.(Chithra et al., 1998) Glucomannan, a mannose rich polysaccharide and gibberellins, a growth hormone, interacts with growth factor receptors on the fibroblast, thereby stimulating its activity and proliferation which forms increased collagen synthesis after topical and oral A. vera administration. This causes accelerated wound contraction leading to

Increased breaking strength of resulting scar tissue.

Immune booster

It may have a direct inhibitory effect on microbes and also selectively modulates cells of the immune system (Chithra et al., 1998). Alovera contains antiseptic agents such as lupeol, salicylic acid, urea nitrogen, cinnamonic acid, phenols and sulfur. They all have inhibitory action on fungi, bacteria and viruses. Cellular cytotoxicity: Human embryonic kidney cells were utilized to determine the effectiveness of alovera gel on cellular longevity. The cellular death rate was found to be reduced by 2/3rd when cultured with alovera gel.

Antimicrobial

The comparative antimicrobial activities of the gel of alovera were tested against Staphylococcus aureus and E.coli. Methanol was used for the extraction of the gel. Antimicrobial effect was measured by the appearance of zones of inhibition.

Anti-inflammation

Evaluated the topical anti-inflammatory activity of alovera. concluded that small amounts of A. vera given topically will inhibit inflammation. Alovera inhibits the cyclogenase pathway and reduces prostaglandin E2 production from arachidonic acid. Recently, the novel anti-inflammatory compound called C-glucosylchromone was isolated from gel extracts**.(**George et al., 2009)

Cancer treatment

Aloe-emodin induces apoptosis in T24 human bladder cancer cells. Investigated anticancer effect of Aloe-emodin (1, 8-dihydroy- 3-[hydroxymethyl]-anthraquione) in the T24 human bladder cancer cell line by studying apoptosis regulation. Aloe-emodin is purified from alovera leaves, has been reported to have antitumor activity. An induction of glutathione S-transferase and an inhibition of the tumor promoting effects of phorbol myristic acetate has also been reported which suggest a possible benefit of using Aloe gel in cancer treatment.(George et al., 2009)

Bleaching

Alovera when added to toothpaste has bleaching property for the teeth. Denture patients with sore ridges and illfitting dentures can benefit as fungus and bacterial Decontamination reduce the inflammatory irritations.(George et al., 2009).

Moisturizing and anti-aging effect

Alovera has rejuvenating action. The mode of action of polysaccharide is that they act as moisturizers, stimulates the fibroblasts to replicate faster and smoothens skin. Fibroblasts produce collagen and elastin fibers, so the skin becomes more elastic and less wrinkled. Its moisturizing effects have also been studied in the treatment of dry skin associated with occupational exposure. The study concluded that alovera gel gloves improved the skin integrity, decreased appearance of fine wrinkles and decreased erythema.(West and Zhu, 2003) .

2.7.3 Drawback of application of ZnO in textile finishing:-

ZnO nano-particles have shown some advantages when compared to other inorganic nano-particle, such as lower cost, white appearance(Vigneshwaran et al., 2006) .and UV-blocking property (Becheri et al., 2008b) .ZnO powders can absorb infra-red light and infra-red electromagnetic wave with 5–16.68 dB in the range of 2.45–18GHz ZnO is also used to reinforce polymeric nano-composites (Vigneshwaran et al., 2006). It has been showed good properties, enhancement wear resistant phase and anti-sliding phase in composites as a consequence of their high elastic modulus and strength(Dastjerdi and Montazer, 2010).

The bulk ZnO and micro ZnO, is safety to use in application of textile finishing. But in Nano size ZnO is become more dinger, especially in textile finishing, because low stability of Nano particles on surface of textile led to release some particles which touch the human skin and interred to body membrane ,stable there and wait the UV light to make oxidation and attack human cells to make huge damage surface . also aggregate of nanoparticles on textile surface lead to create fabric surface with nonhomogeneous properties, and make release particle high in some point which have aggregate more particles than those how have low aggregate. The scientist tried to fix these problem by using plant extract (Alovera, fun-Greek ,fruit acid) to act as stabilizer agent to enhance the attach between textile surface and ZnO particles.

Qing Zhou and et.al were synthesized Zinc oxide (ZnO) micro/nano-composites with the natural capping agent: aloe gel extract (AGE) instead of traditional chemical capping agent. The results showed that ZnO/AGE MNPs with smaller particle size were homogeneous and spherical shape crystal and assigned, to the ZnO phase .the treated fabric with those nanoparticles (ZnO/AGE MNPs) has shown smooth surface when it was compered to untreated. After being washed for 20 times, the treated fabric was still UV resistance. (Zhou et al., 2017)

2.7.4 Advantages and Disadvantages of textile nano finishing:-

Advantages:-

- Single step method
- Reduced time, chemicals and energy
- Cost-effective
- Physical and/or chemical interactions
- Higher durability
- Low toxicity

Dis advantages:-

- Smaller sized nanoparticles in low precursor content
- Positive influence on other properties such as enhanced Tensile strength
- High-temperature calcination for crystallizations
- Smaller sized nanoparticles in low precursor content
- Long time calcination at low temperature
- Process at very low or high pH
- Uncontrolled shape and size in high precursor content
- Low level of deposition causing more nanoparticles remain in bath

CHAPTER THREE

Materials, Methods and Characterization

3 .1 Materials:

3.1.1 Fabric

Plain weave (1/1) 100% cotton fabric with 150 GSM, 20 Ne warp and weft count, 75 ends/inch and 54 picks/inch was used in this research work.

3.1.2 Chemicals

Zinc acetate-dehydrate (CH_3 COO $)_2$ Zn.2H_2O from WAKO Japan ,caustic soda from Mumbai ,India, concentration (2M) and citric acid from Swiss . Aloevera (Aloe-emodin1,8-dihydroxy-3(hydroxyl-methyl)-9,10 anthracene dione) was taken from plants at home (Khartoum-Sudan). Methanol (CH_3OH) from Darmstadt, Germany, was used for diluted Alovera gel.

3.2 Methods: -

3.2.1 Alovera (barbadensis, asphodelacease) extraction

Mature and fresh leaves of Alovera (aloe barbadensis miller, belong to asphodelacease family) was washed in the running tap water for 5 min, then was dissected longitudinally using a sterile knife and cutting in cub shape then mixed with kitchen blender to get Alovera gel. (Gediya et al., 2011)

Fig (3.1): Alovera leaf gel

Fabric modified with ZnO (F –ZnO)

Fabric-ZnO was prepared via a sol–gel process using zinc acetate anhydrite (Zn $(Ac)_2.2H_2O$) as a precursor material as follow:

One gram (1 g) of fabric was immersed in 150 ml of deionized water and mixed with 0.5 g of Zn $(Ac)_2.2H_2O$ pre-dissolved in 5 ml methanol (5% w/v). A concentrated sodium hydroxide solution (2 M) was added drop-wise to the mixture to keep the ph above 10 during the reaction. The mixture was heated to 70°C under vigorous stirring until a milky white solution was obtained. There after a milky solution was heated for a further (2 h) under the same temperature. Fabric was removed, and squeezed, washed by distilled water and dried at room temperature.

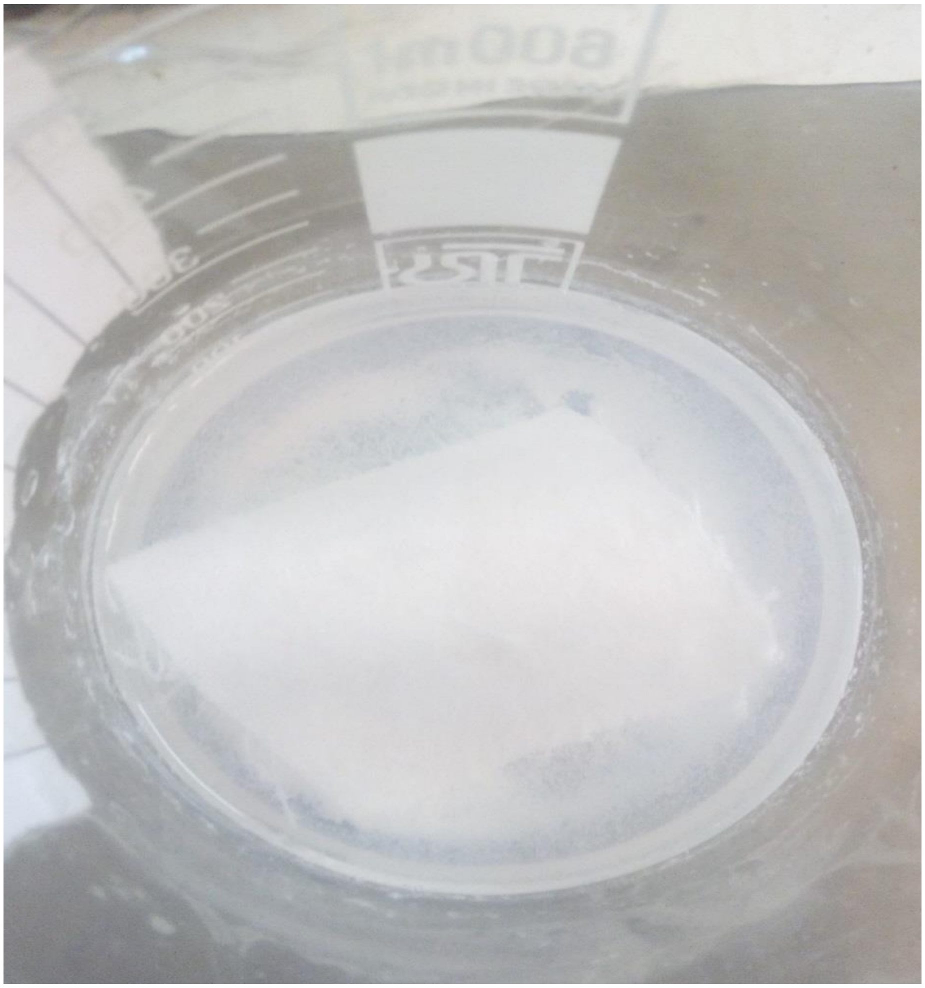


Fig (3.2): The modified fabric with ZnO (F-ZnO)

Fabric modification with Alovera and ZnO (F- A –ZnO)

30 ml of Alovera gel diluted by 70 ml of methanol ,1 g of fabric was impeded in solution, for 24 h then fabric was separate the fabric and dried at room temperature 0.5 g was suspended in 150 ml of deionized water and mixed with 0.5 g of $Zn(Ac)_2.2H_2O$ pre-dissolved in 5 ml methanol (5% w/v). A concentrated sodium hydroxide solution (2 M) was added drop-wise to the mixture to keep the pH above 10 during the reaction. The mixture was heated to 70°C under vigorous stirring until a milky white solution was obtained. Thereafter the solution was heated for a further

(2 h) under the same temperature. then the fabric, removed, squeezed and washed with distilled water and dried at room temperature.

Fig (3.3): The modified fabric with Alovera (F-A-ZnO)

Fabric modification with citric acid and ZnO (F-CI-ZnO)

(2g) of citric acid dissolved in (100 ml) distilled water, and (1g) of fabric was immersed in solution for (30 min). Then, the fabric was removed, rinsed and dried at room temperature. The dried sample, was placed on oven at 120 C^0 for (10min), to dehydration of citric acid. then, sample was immersed in (150 ml) of deionized water and mixed with(0.5 g) of Zn(Ac)2.2H2O, pre-dissolved in (5 ml) methanol, (5% w/v). Concentrated sodium hydroxide solution (2 M), was added drop-wise to the mixture to keep the PH above 10 during the reaction, then mixture was heated to (70°C) under vigorous stirring until a milky white solution was obtained. Then the solution was heated for (2 h) under the same temperature, Then fabric was taken out squeezed, rinsed , washed by distilled water, and dried at room temperature.

Fig (3.4): the modified fabric with Citric (F-CI-ZnO)

3.3 Characterization:-

3.3.1 Fabric test

Cotton fabric was tested under ban and counted number of weft and warp per inch were found 75 ends/inch for warp and 54 picks/inch for weft. with weight of 150 GSM per meter square.

Fourier Transform Infra- Red (FTIR)

FT-IR stands for Fourier Transform Infra- Red , the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. off This makes infrared spectroscopy useful for several types of analysis. Fourier Transform Infrared (FT-IR) spectrometry was developed in order to overcome the limitations encountered with dispersive instruments. The main difficulty was the slow scanning process. A method for measuring all of the infrared frequencies simultaneously rather than individually, was needed. A solution was developed which employed a very simple optical device called an interferometer. The interferometer produces a unique type of signal which has all of the infrared frequencies "encoded" into it. The signal can be measured very quickly, usually on the order of one second or so. Thus the time element per sample is reduced to a matter of a few seconds rather than several minutes. Fourier transform infrared spectroscopy is preferred over dispersive or filter methods of infrared spectral analysis for several reasons:

- It is a non-destructive technique
- It provides a precise measurement method, which requires no external calibration
- It can increase speed, collecting a scan every second

• It can increase sensitivity – one-second scans can be co-added together to ratio out random noise

• It has greater optical throughput

• It is mechanically simple with only one moving part

The normal instrumental process is as follows

1. **The Source:** Infrared energy is emitted from a glowing black-body source. This beam passes through an aperture which controls the amount of energy presented to the sample (and, ultimately, to the detector).

2. The Interferometer: The beam enters the interferometer where the "spectral encoding" takes place. The resulting interferogram signal then exits the interferometer.

3. The Sample: The beam enters the sample compartment where it is transmitted through or reflected off of the surface of the sample, depending on the type of analysis being accomplished. This is where specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed.

4. The Detector: The beam finally passes to the detector for final measurement. The detectors used are specially designed to measure the special interferogram signal.

5. The Computer: The measured signal is digitized and sent to the computer where the Fourier transformation takes place. The final infrared spectrum is then presented to the user for interpretation and any further manipulation.

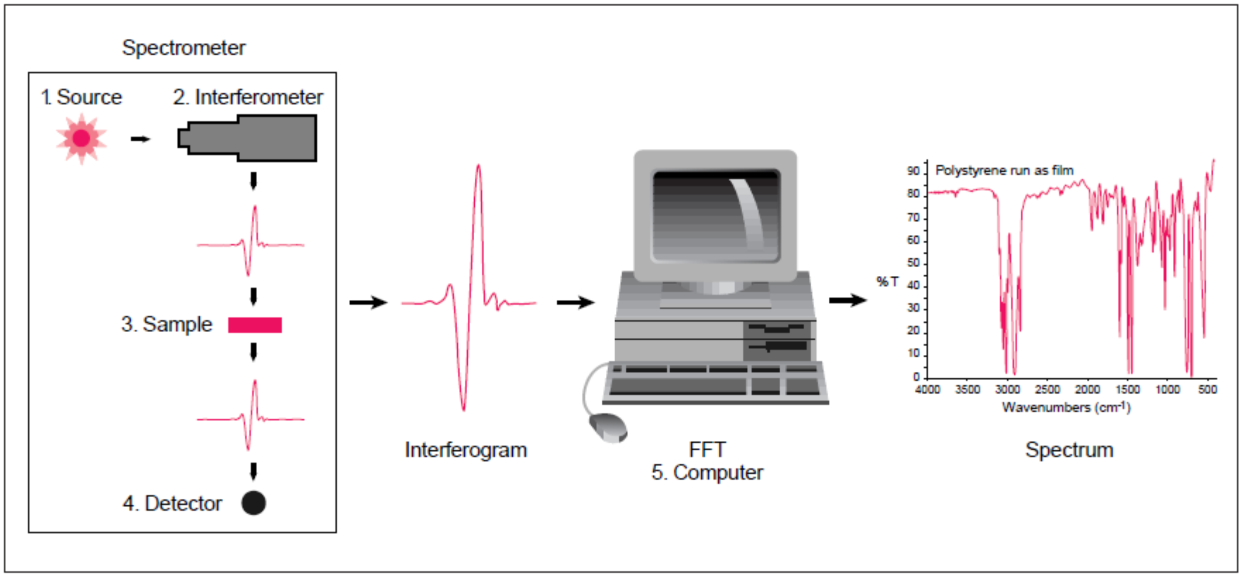


Fig (3.5): FTIR Instrument

Scanning Electron Microscope (SEM):

Scanning Electron Microscope – SEM is based on the scanning of the specimen surface by an electron fascicle and the analysis of the signal (electromagnetic particles and waves). resulting from the interaction between the primary fascicle and the specimen ,The depth at which information on the specimen is obtained ranges between 1 nm (Auger electrons) and 5 μ m (characteristic X radiation). With SEM the contrast may be of the following types: topographic contrast, atomic number contrast, magnetic contrast, etc.

– Maximal resolution is about 25 Å or 5 Å (field emission beam);

– Magnifying by 20–500000 times

– Atoms or molecules cannot be visualized

– Specimens must have good electrical conductivity. Those without electrical conductivity must be covered by a metallic layer prior to examination or examined in reduced vacuum in order to prevent electrostatic charge.

Preparation of specimens is relatively easy i.e. Is based on the effect of the structural characteristics of the material to be investigated on the passage of an accelerated electron fascicle through a very thin specimen (electrons transmitted through the specimen, position). The contrast of the image obtained is given by the difference between the absorption coefficients of the different specimen points.

– Maximal resolution is under 0.1 nm at × 106 magnitude. Thus atoms or molecules can be visualized).

– It may be provided with an X-ray spectrophotometer for local quantitative (composition) chemical analysis.(Vida-Simiti et al., 2004)

Scanning electron microscopy and Electron diffraction (SEM-EDS)

EDS makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle, though not all instruments are equipped for 'light' elements ($Z < 10$). Qualitative analysis involves the identification of the lines in the spectrum and is fairly straightforward owing to the simplicity of X-ray spectra. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration standards of known composition. By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line, element distribution images or 'maps' can be produced. Also, images produced by electrons collected from the sample reveal surface topography or mean atomic number differences according to the mode selected. The scanning electron microscope (SEM), which is closely related to the electron probe, is designed primarily for producing electron images, but can also be used for element mapping, and even point analysis, if an X-ray spectrometer is added. There is thus a considerable overlap in the functions of these instruments.

Fig (3.6): SEM and EDS Instrument

3.3.2 Physical tests

Crease recovering

The ability of fabrics to resist the formation of crease or wrinkle when lightly squeezed is termed as crease resistance of the fabric is associated with the fabric stiffness(Akbarzadeh et al., 2012) .Cotton though is comfortable to wear; it has very poor crease recovery angle (CRA). Cotton fabrics are most prone to crease formation, since their ability to recover from the applied deformation is poor and has been explained in the mechanism of crease formation. The crease recovery of the fabrics have been tested using the Shirley crease recovery tester as it shown in figure(3.7), and the value reported in this instrument was crease recovery angle (CRA) (KANADE and KORANNE, 2015).

The instrument consists of a circular dial which carries the clamp for holding the specimen (see figure (3.7)) directly under the center of dial is a knife edge and an index line for measuring the recovery angle .the scale of the instrument is engraved on the dial.

A specimen was cut from the fabric with a templet, 2 inch in length by 1 inch in width. It is carefully creased by folding in half .placing it between two glass plates, and adding a 2 kg weight. After (1 min) the weight is removed, and the specimen transferred to the fabric clamp on the instrument, and allowed to recover from the crease. As it recovers the dial of the instrument, is rotated to keep the free edge of the specimen in line with the knife-edge. At the end of the time period allowed for recovery usually (1 min) the recovery angle in degrees is read on the engraved scale. Warp and weft way recovery are reported separately to the nearest angle from the mean values of ten tests in each direction .the load time of creasing and recovery time may be altered to suit particular cases. As for most tested, the specimens should be conditioned and tested in standard testing atmosphere. a random sample should be taken but the selvedges ,piece ends and creased or folded regions should be avoided.(Russell, 1994).

Fig (3.7): the’ Shirley’ crease recovery instrument (CRA)

Shrinkage test:

Shrinkage test is a parameter of testing textile fabrics to measure changes in length and width after washing (laundry). Shrinkage failing materials are dimensionally unstable, and they can cause de- shaping and deforming of the garments or products made out of those materials. Fabric should be tested at various stages, but the important one is the testing before cutting the fabric into further sewn products and after cutting and sewing prior, to supplying the products to buyers and end users. It is a necessarily required parameter of quality control, to ensure the sizes of the products to avoid any complaints regarding deformation or change in dimensions after domestic laundry (Akbarzadeh et al., 2012). The tests (Becheri et al., 2008a) are conducted with provided specifications of buyers imitating the same conditions like washing cycle time, temperature and water ratio and fabric load and sometimes top loading and front loading washing machines are chosen to authenticate the test and assurance of the results. This procedure provides standard and alternate home laundering conditions using an automatic washing machine. While the procedure includes several options, it is not possible to include every existing combination of laundering parameters. The test is applicable to all fabrics and products suitable for home laundering.

Types of shrinkage

Shrinkage is a change in dimensions across the length and width of the fabric after washing, usage and when exposed to relaxing of fabrics. Mainly shrinkage is of two types.

1-Contraction: Any noticeable decrease in dimensions is known as Contraction (minus) shrinkage.

2-Expansion: Any noticeable increase or expansion in dimension is known as Expansion (plus) shrinkage.

Causes of shrinkage:

1-Composition and properties of the fibers

Composition and content determine the type of fibres. Natural fibres shrink more than synthetic fibres (fibers made by human). Synthetic fibres are more stable due to their crystalline and thermoplastic nature. They do not shrink, whereas natural fibres are more prone to shrink because of more amorphous region in their fibre structure, which allows more absorption of water, swelling of fibres, and increased lubricity increases the shrinking tendency. Blended fabrics normally synthetic and natural are also considered more stable. Structure of the fabric/ knit or weave loose and tight structure. This factor also plays a vital role in the shrinking of the products. The products, which are loosely woven or knitted are prone to shrink more and tightly knitted and woven products, are more stable. The main reason is that knitted fabrics shrinkage is because they are made by interloping the yarn which is comparatively a loose and flexible structure whereas woven are considered more stable and less sensitive to shrinkage.

2- Finishing applications and procedures

Fibers to fabric conversion implies lots of mechanical tensions and forces during manufacturing, which includes following steps for fibre to yarn conversion with spinning then fabric with weaving, knitting. When the products are immersed in water, the water acts as a relaxing medium and all stresses and strains get relaxed and try to come back to its original relaxed state. Even after finishing with sophisticated finishing machines some residual shrinkage remains, which is carried forward to the garment stage. This residual shrinkage may cause deformity or deshaping of the products after domestic laundry. There are certain acceptance limits of shrinkage levels for every product. Abnormal shrinkage levels are considered a non-conformity to quality standards.

Test methods the different test methods are used as per the final destination of the product like in Europe, U.S.A., etc. and expected washing or laundry methods in practice over there. Mainly I.S.O. and AATCC standards are used for shrinkage testing. There are few brands, which are customizing the test method as per their quality norms.

3-Test Method(s):-

Shrinkage measuring template, scale and marker

· AATCC Test Method 135

· AATCC Test Method 150

· ISO 6330

· CAN/CGSB 58

AATCC Test Method 135, dimensional change of fabrics after home laundering Scope: Determines the dimensional changes of garments when subjected to home laundering procedures used by consumers. The method is for fabric not yet made into a garment. A sample is marked with benchmarks before home laundering. Then it is laundered 3 times total, then the benchmarks are measured again. Before and after laundering benchmarks are compared. AATCC Test Method 150, Dimensional Change of Fabrics after Home Laundering Scope: Determines the dimensional changes of garments when subjected to home laundering procedures used by consumers.

Abrasion

The crock meter is a relatively simple rub tester commonly used to determine the amount of color transferred from textile materials to other surfaces by rubbing. This instrument has also been utilized to conduct smear or abrasion resistance tests on images produced by a printer or copier. For paints and coatings, the crock meter is useful when evaluating the change in gloss due to rubbing, scuffing or marring. A test sample is clamped to the instrument base and a square of standard crocking cloth is fixed to a 16mm diameter, acrylic-rubbing finger. The finger rests on the sample with a pressure of 900 grams force and traverses a straight path approximately 100mm long with each stroke of the arm.

Fig (3.8): Crock meter Instrument

Tensile test

Tensile test is most commonly applied test method for analyzing the mechanical properties of fabric materials. Although the direction of applied force is always in tension. BELLSTONE- HI-TECH INTERNATIOAL made in India, it is use to examine tensile force. Specimen with 30 cm long and 5 cm width is gripped in the tensile grip jaws of bellstone tensile test instrument ,during this test ,tensile force applied on the fabric specimen until it rupture ,the mechanical properties analyze include the force at rupture and the elongation .

Fig (3.9): Tensile test instrument

3.3.3 Antimicrobial test:

Purpose and Scope

This test method provides a quantitative procedure for the evaluation of the grade of antibacterial activity. Assessment of antibacterial finishes on textile materials is determined by the grade of antibacterial activity intended in the use of such materials. If only bacteriostatic activity (inhibition of multiplication) is intended, a qualitative procedure which clearly demonstrates anti-bacterial activity. As contrasted with lack of such activity by an untreated specimen may be acceptable.

However, if bactericidal activity was intended or implied , quantitative evaluation is necessary. Quantitative evaluation also provides a clearer picture for possible uses of such treated textile materials.

AATCC 147 is a qualitative antimicrobial test used to detect bacteriostatic activity on textile materials. This test determines antibacterial activity of diffusible antimicrobial agents on treated textile materials. It provides a qualitative zone of inhibition around the treated fabric. The size of the zone of inhibition and the narrowing of the streaks caused by the presence of the antibacterial agent permit an estimate of the residual antibacterial activity after multiple washings.

Antimicrobial activity of the ZnO-coated fabrics was studied with standard methods: (American Association of Textile Chemists and Colorists) (AATCC) (Amirbayat and Alagha, 1996), The former method shows bactericidal activity. (E coli, Gram-negative bacterium) and Staphylococcus aureus (S. aureus, Gram-positive bacterium) were used as model challenge microorganisms. Nutran agar was prepared as a solid media, modified fabrics was fixed on surface of media at 37°C for 24 h. Then, was observed loop in each plate, and take photoset for plate. (Bashari et al., 2018)

CHAPTER FOUR

Result and discussion:-

4.1 Fourier-transform infrared spectroscopy (FTIR):

ZnO nanoparticles was applied on cotton fabric to improve anti-bacterial, physical and mechanical properties .it was applied with selected natural cross linking agents, the treated fabric samples was investigated by Fourier-transform infrared (FT-IR) spectroscopy.

FTIR has proved to be one of the most useful methods to study the chemical composition of material. Furthermore FTIR can provide researchers with further information on the super-molecular structure. FTIR can also be use to determine the chemical compositions of native natural fibers and the modified natural fibres. In this work (FTIR-8400S) SHIMADAZU – JAPAN instrument, was used to investigate chemical compositions of samples, which showed at FTIR spectrum. Analysis results was showed new peaks were found in treated samples (F-ZnO),(F-A-ZnO), and (F-CI-ZnO) when were compared with the control sample peaks, which its cellulose. FTIR transmission spectrum of cellulose showed OH stretching vibration regions between 3000 and 3500 cm^{-1}, The OH stretching region always covers 3-4 sub-peaks and these sub-peaks cannot be determined in the original data set.(Fan et al., 2012)

Investigation result of sample (F-A-ZnO) was showed, the absorption peaks at 1400, 1500 and 3000 cm^{-1} which are characteristic of peaks in alovera and are due individually, to the C=O stretching of carboxylic ester ($–COOCH_3$) and the asymmetrical and symmetrical $–COO^-$ of carboxylic acid (–COOH) groups.(Ali et al., 2014)

FTIR spectrum of sample (F-CI-ZnO), in addition to cellulose peaks, showed a new peaks between 1500 and1845 cm^{-1} and it related to C=O stretching vibration peak.(Shi et al., 2008) and it is probably a coalescence peak which is caused by the ester bond and carboxyl. The new observed peaks is resulted of treated sample with CI. This confirm that the modification was achieved successfully.

The sample which treated by ZnO nanoparticles without using cross linking agent showed new peaks in modified fabric, to detect of this peaks farther more(SEM) had been used and it was definitely confirmed presence of ZnO nanoparticles in samples.

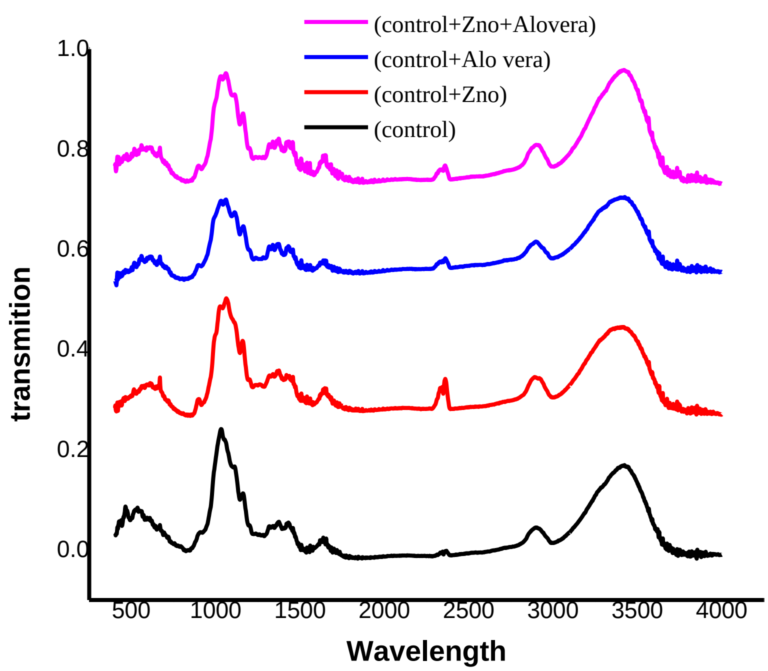


Fig (4.1): FTIR of (Control, Control + ZnO, Control +Alovera and Control + ZnO +Alovera)

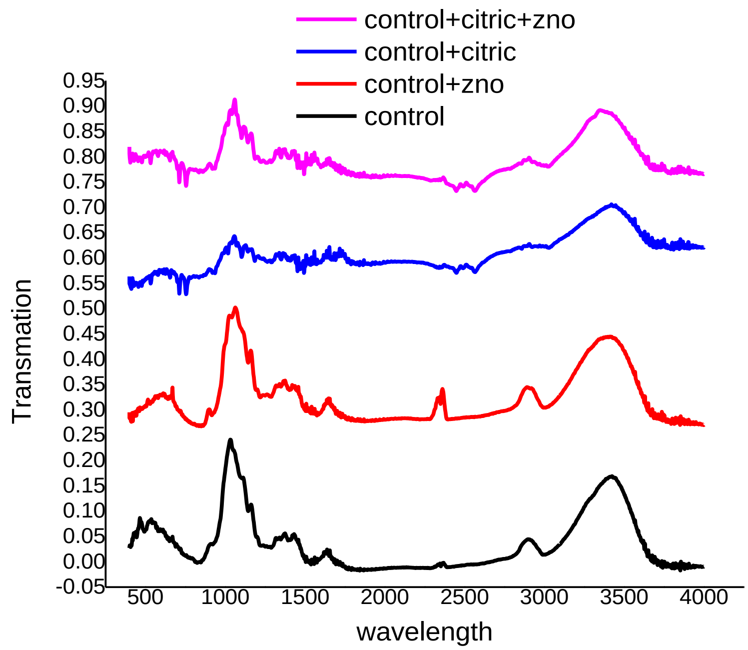


Fig (4.2): FTIR of (Control, Control + ZnO, Control+ Citric, Control +Citric +ZnO)

4.2 Scanning Electron Microscopy (SEM)

Morphological changes of the cotton fabric treated by solution of zinc acetate dehydrate can be clearly seen from the scanning electron microscopy (SEM) as shown in (Figure (4.3), (4.4), (4.5), (4.6)). The presence of ZnO nanoparticles on fibers' surface is clearly distinguished. As it can be seen homogenous and nonhomogeneous ZnO nanoparticles, were formed, and distributed on the fiber surface. In fig (4.3), (F-ZnO) observed amount of ZnO nanoparticles were synthesized on the surface of the fiber and this was approved by tested via (SEM), as was obvious in fig(4.3). This evidence to successfully synthesis ZnO nanoparticles on fiber by sol- gel methods which we used to synthesis ZnO nanoparticles , they were synthesized with average of diameters size (138.85 nm), this seen clearly in fig(4.4), which was tested under magnification of 500 nm. (F-A-ZnO) at magnification (2micro), the results was showed, huge amounts of ZnO nano particles aggregated on the surface of the fabric, and a few amount dispersed randomly at whole fibers surfaces area, this due to non-homogenous hydroxlics groups, presented by alovera which led to catch a huge amounts of ZnO nanoparticles in some positions and few amount in other positions. a unique Alovera structure tend to catch whole amount of ZnO nano particles were synthesis in reactor approximately all amount of ZnO nano particles were holded on fabric surface, which it mean alovera structure acts as good crosslink between fiber and nanoparticles , confirmed the stability during repeated laundry. By average diameters (59.2 nm) via magnification (500 nm), the ZnO nanoparticles were synthesized on surface of fibers this showed clearly in fig (4.5).

ZnO nano particles clustering on the surface of fibers, like bundle, at different size with average diameters (70.6 nm) under magnification (2µm), as it is shown obvious in fig (4.6). ZnO nano particles were clustering but was distributed in homogeneous miner on the surface of the fibers which is refer to homogeneous distribution hydroxilic groups in citric acid structure, which has ability to catch amount of ZnO nanoparticles with homogeneous distribution. Alovera structure showed high ability of catch ZnO nanoparticles with smallest dimeters average (59.2nm), which led us to infer Alovera have good cross-linking properties.

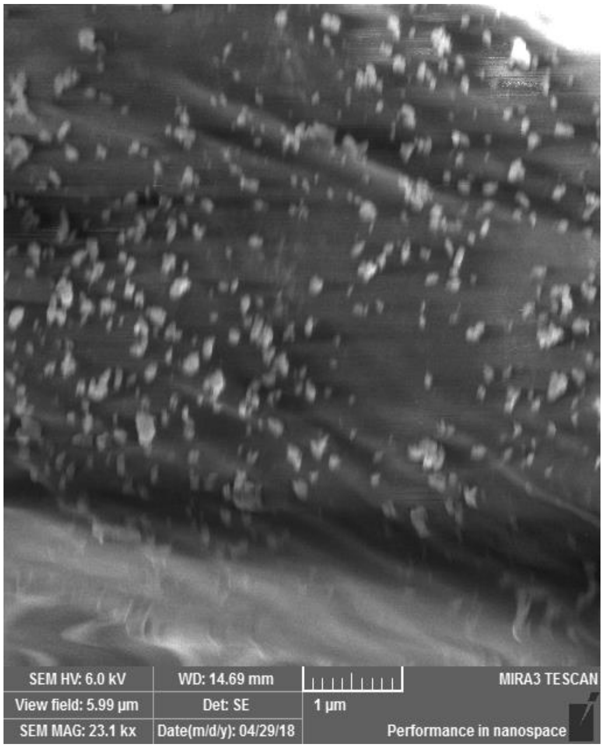


Fig (4.3): SEM of fabric modified with ZnO at magnification (1µm)

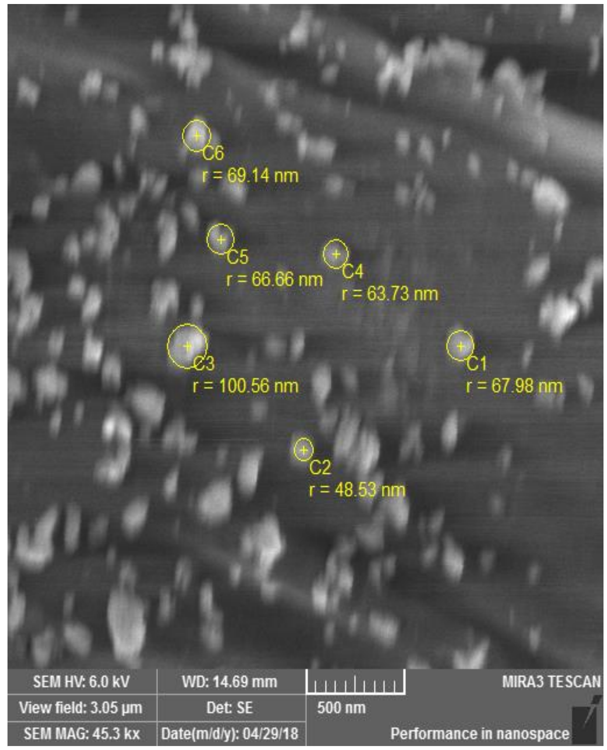


Fig (4.4): SEM of fabric modified with ZnO at magnification (1µm)

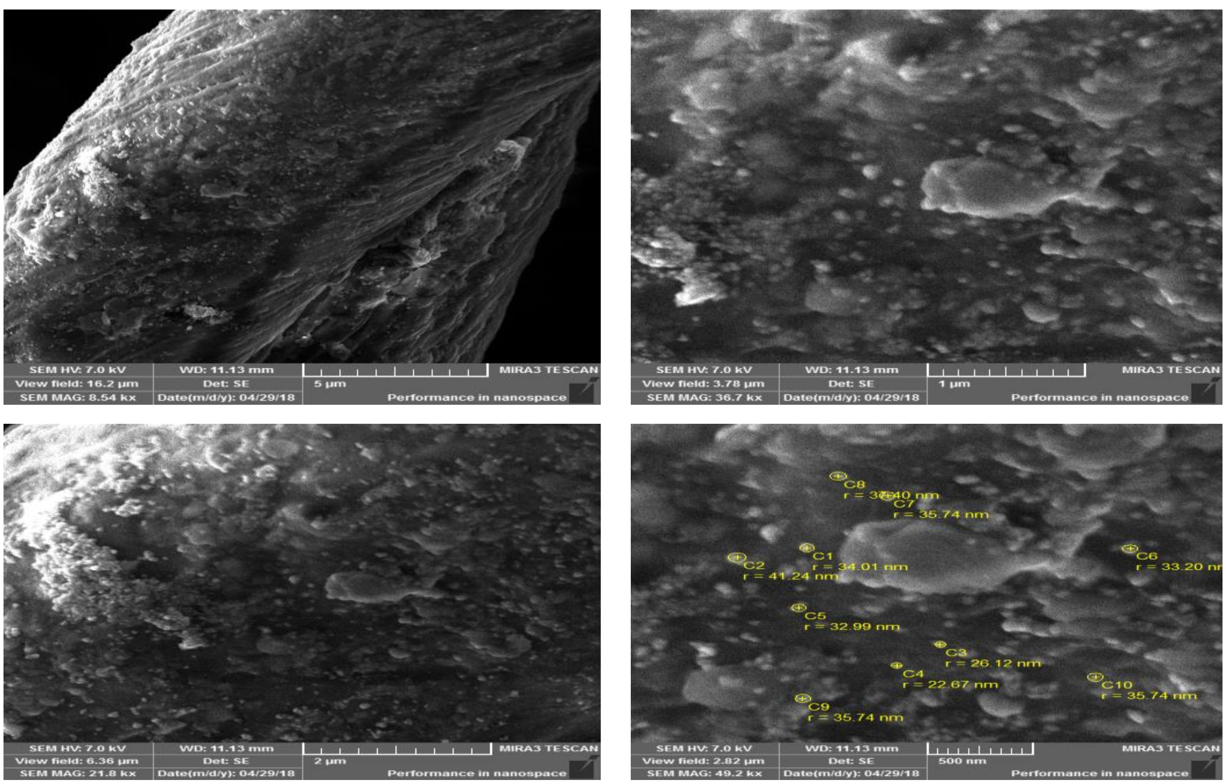


Fig (4.5): SEM of modified fabric with Alovera at magnification (5µm, 1µm, 2µm, 500nm)

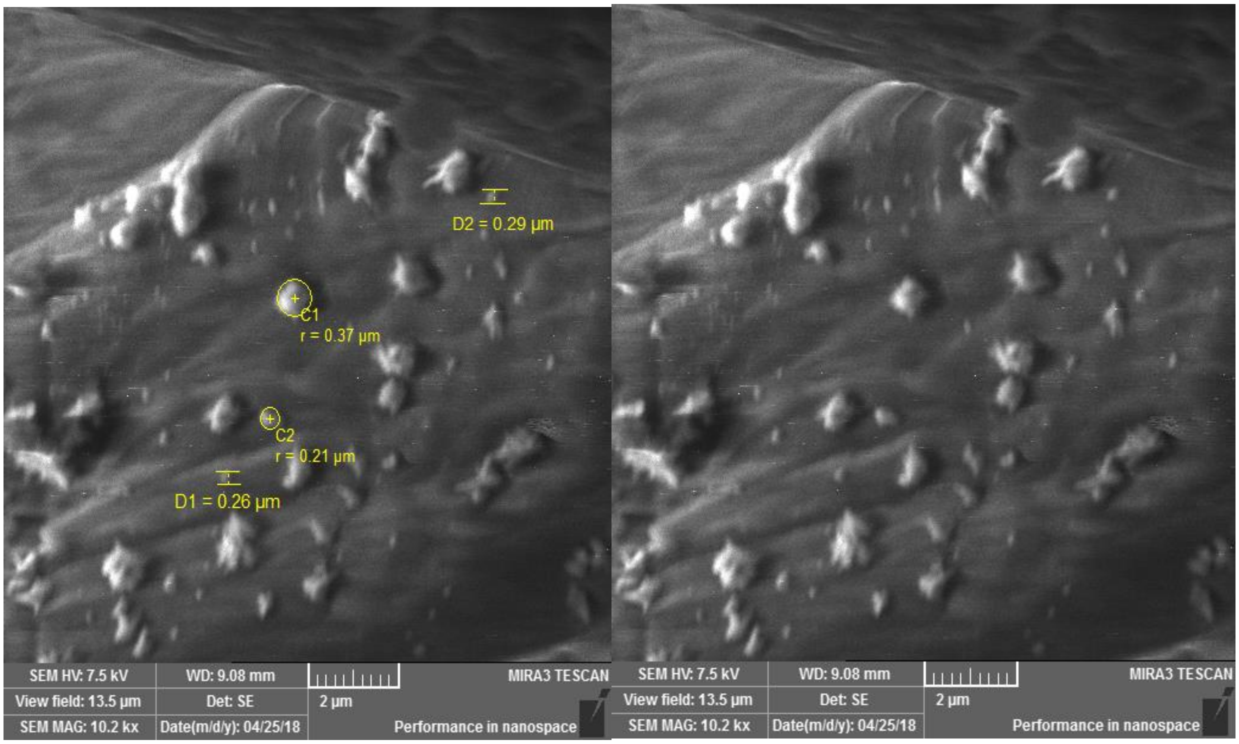


Fig (4.6): SEM of fabric modified with citric at magnification (2µm)

4.3 EDS TEST:

EDS measurements were carried out for element detection of the fabric surface. The representative EDS patterns are shown in Figure (4.7). Peaks at about 9.5 keV are characteristic for O, Na and Zn signals are located at about 0.4,1.0,1.0, 8.6, and 9.6 keV (Shateri-Khalilabad and Yazdanshenas, 2013). The peaks of Si and Ca were observed at about 1.8 ,1.0 and 3.9 keV, which are from the lap environment. The EDS results reveal that the prepared nanostructures are certainly composed of Zn and O. The concentration of Zn element was 3.3 wt. % and the ZnO quantity was 0.5 g/g, when was treated samples (F-ZnO),(F-A-ZnO) and (F-CI-ZnO) It should be noted that the ZnO content determined by EDS is always different from the actual amounts on materials. This discrepancy is experienced because although the penetration depth of EDS is about 500 nm, most ZnO particles are localized on the fiber's surface of cotton fibers.

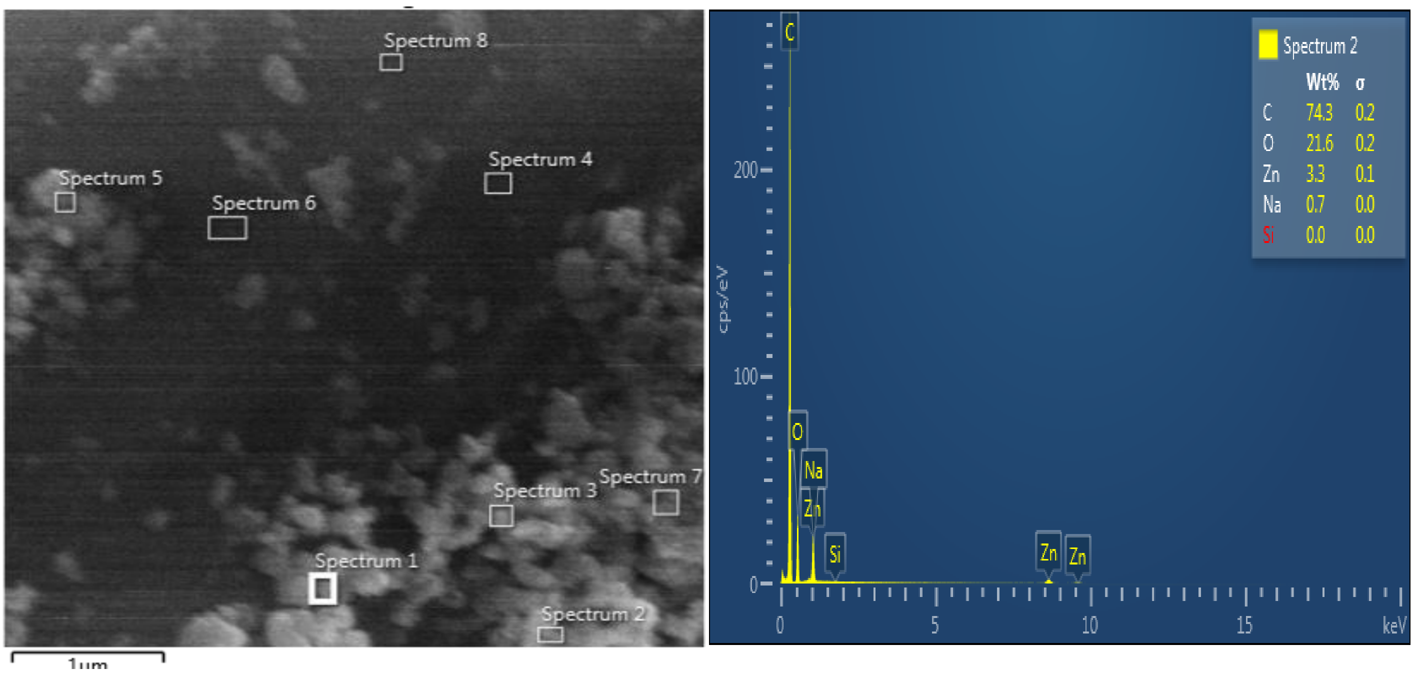


Fig (4.7): EDS of fabric modified with ZnO (F-ZnO)

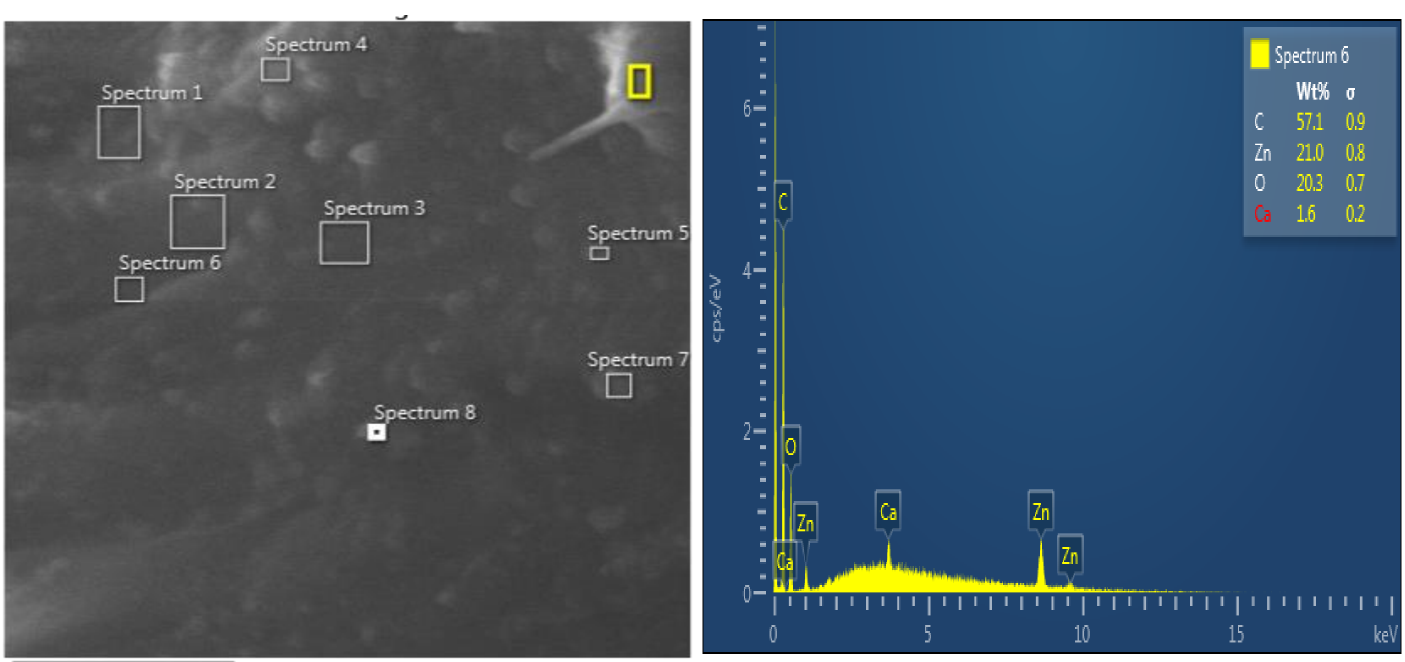


Fig (4.8): EDS of fabric modified with ZnO and alovera (F-A-ZnO)

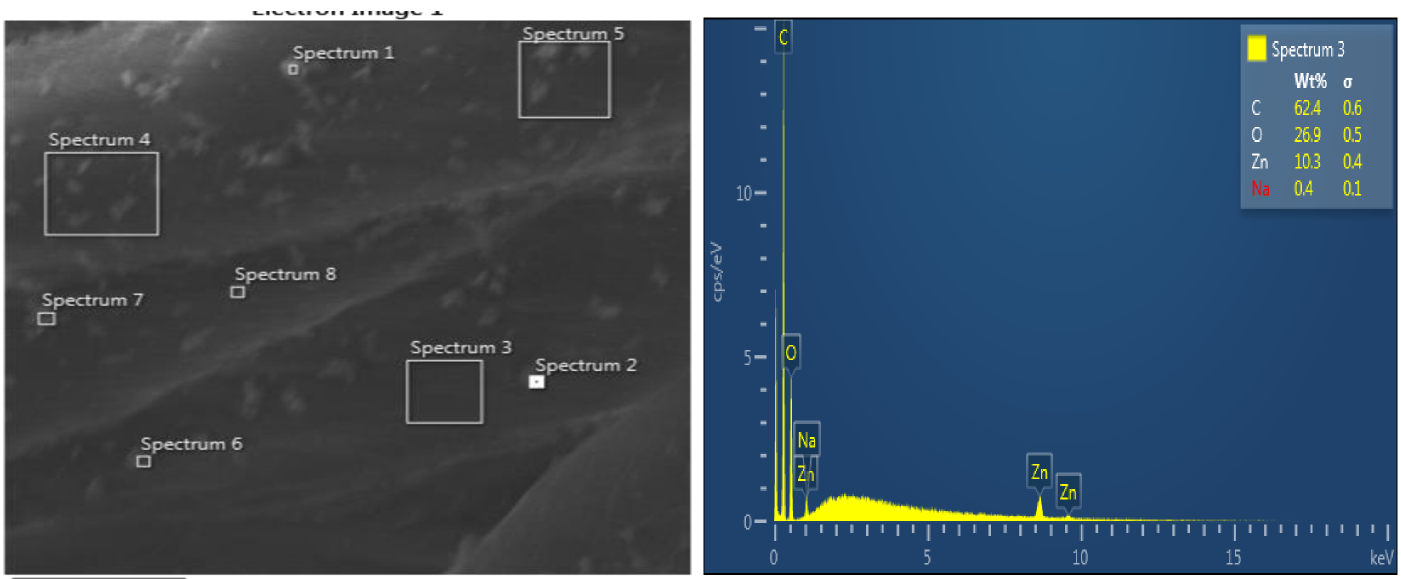


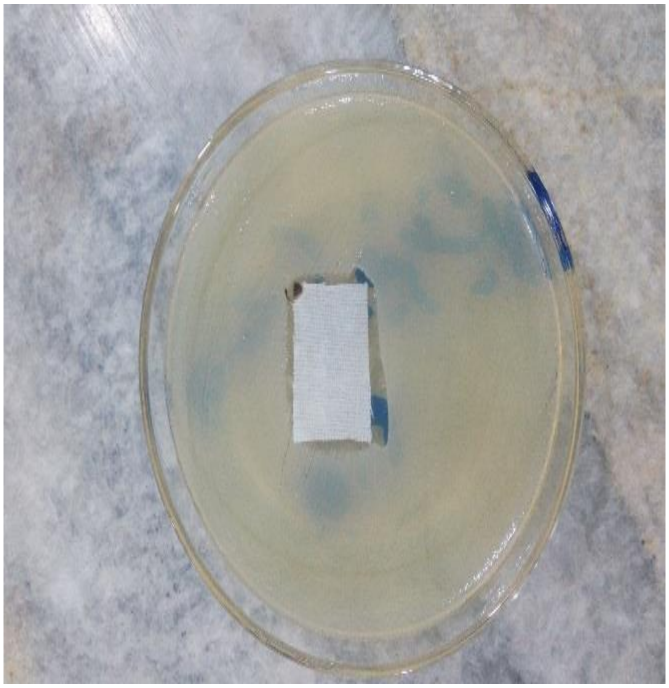
Fig (4.9): EDS of fabric modified with ZnO and citric (F-CI-ZnO)

4.4 Anti-bacterial test:-

Fabrics are an excellent medium for the growth of microorganisms when the basic requirements such as nutrients, moisture, oxygen and appropriate temperature are present. Natural fibers like cotton are more susceptible to microbial attack than synthetic fibers(Szostak-Kotowa, 2004) . ZnO is preferable than other inorganic forms of zinc because of its lower toxicity and higher efficiency in preventing bacterial.(Vigneshwaran et al., 2006) In the control fabric (figure 10(a)), the growth of both Staphylococcus aureus and E colie was found on the fabric as well as surrounding the fabric sample. Hence, cotton coated with nano-ZnO was evaluated for antibacterial activity.(Perelshtein et al., 2008) cotton fabric was treated by ZnO nanoparticles and investigated anti-bacterial properties against Escherichia coli (Gram negative) and Staphylococcus aureus (Gram positive). The tests were performed under dark conditions agar diffusion method had been used. In this agar diffusion method, two-layered agar plates were prepared. The lower agar layer consisted of 10mL of ordinary agar, whereas the upper layer consisted of 5 mL agar inoculated with bacteria, whereby 1mL of bacteria working solution with a concentration of (1^{-5}*$10^{8)}$ colony forming units per milliliter (CFU/mL) was added per 150mL of agar. Circular cotton specimens, with diameter of 20 mm, were uniformly pressed on the agar, and incubated for 24 h at (37±$1C^{0}$). After incubation, the bacterial effect of the fabrics was assessed by evaluating different parameters: the growth of the bacteria underneath and the presence of at least 1mm of inhibition zone around the specimen. Ideally, no bacterial growth should be observed on the fabric. Thus a ‘‘no growth’’ category represents ‘‘ideal’’ antibacterial efficacy; that is, no bactericide elutes into the bulk of the agar with no bacterial growth on the cotton.(Shateri-Khalilabad and Yazdanshenas, 2013).

4.4.1 Fabric modified with ZnO (F–ZnO):-

To investigate the performance of antibacterial properties anti- microbial test was done before and after washing (5 cycles), The result showed anti-bacterial activity for ZnO nano particles on fabric which observed clearly from zoon which formative around the fabric in bacteria ambient, the activity of ZnO was observed before and after washing fabric. Which was indicator to durability of bactericidal activity even after washing large zoon formative around (**F- ZnO**) in staphylococcus ambient and it was seem organized in whole direction and stable before and after washed in distilled water for 5 cycle ,rinsed and dried. Verses in Ecoli ambient small zoon, non –organized and it seem stable before and after washing .



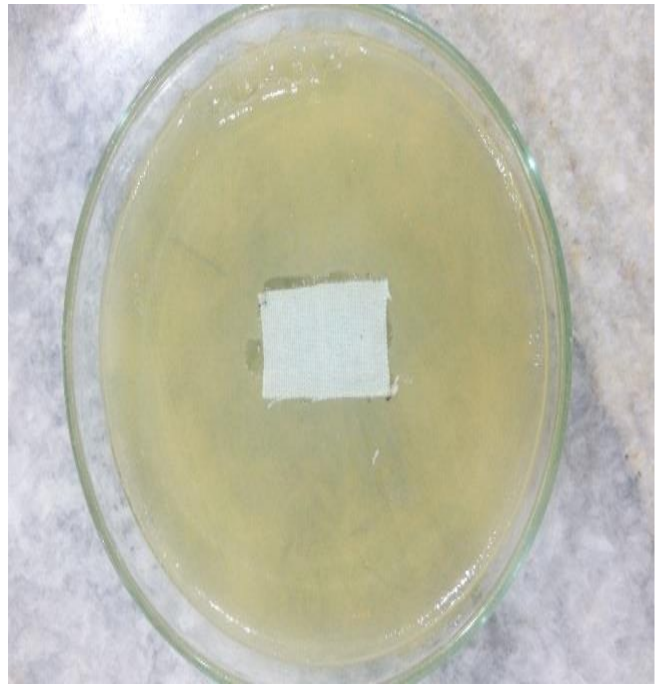
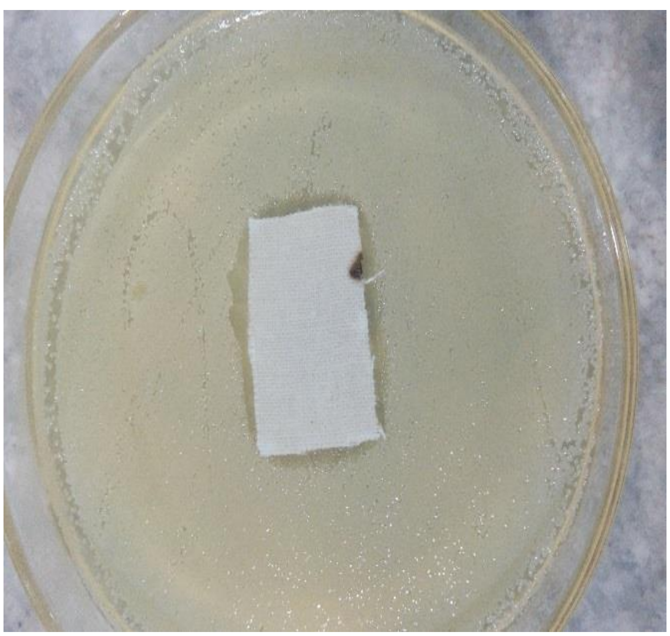


Fig (4.10): The treated fabric with ZnO (St- before washing)

Fig (4.11): The treated fabric with ZnO (Ecoli- before washing)



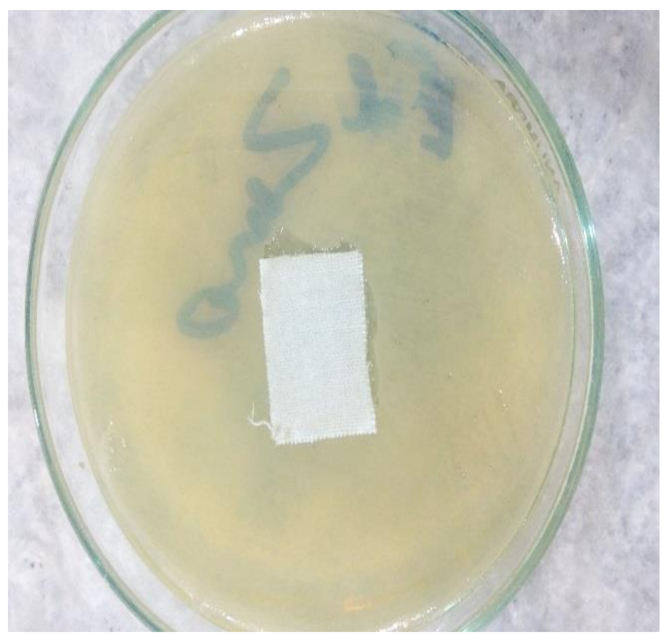


Fig (4.12): The treated fabric with ZnO (St-after

Fig (4.13): The treated fabric with ZnO (Ecoli-after

4.4.2 Fabric modified with (Alovera) and ZnO (F- A –ZnO) :-

Huge and organized zoon formative around the fabric **(F- A –ZnO)** before washed which present a higher bactericidal effect against staphylococcus, small and nonorganized loop formative against Ecoli. This present a good anti-bacterial result of ZnO nano particles against staphylococcus (gram positive bacteria) and acceptable result with Ecoli (gram negative bacteria). After washing 5 times the loop turn to non- organized shape but still huge dimensions with staphylococcus and small with E coli.

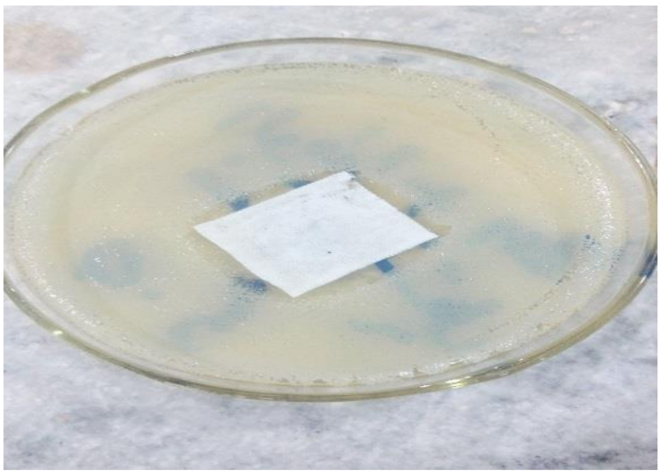


Fig (4.14):(A-ZnO) Treated fabric with(St- before washing)

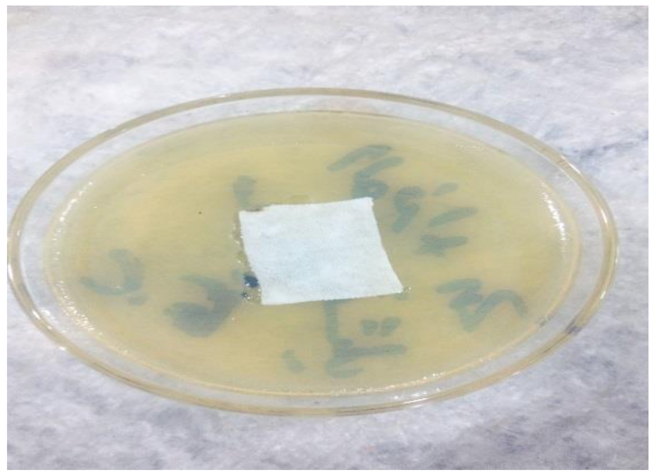


Fig (4.15): (A-ZnO) treated fabric with (E coli before washing)

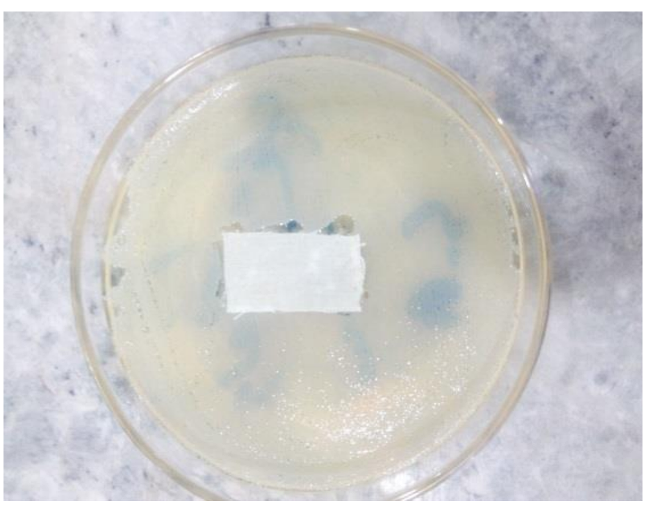


Fig (4.16) :(A-ZnO) Treated fabric with (St-after washing)

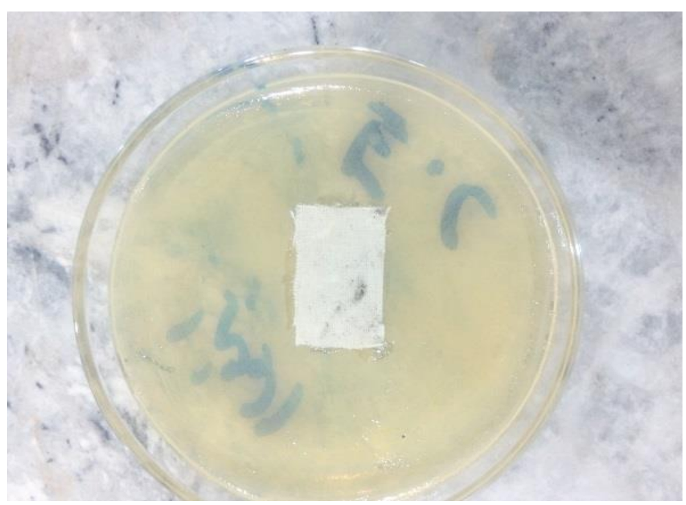


Fig (4.17): (A-ZnO) Treated fabric with E- coli after washing

4.4.3 Fabric modified with (citric acid) and ZnO (F- CI –ZnO) :

zoon with Small organized shape formative around **(F- CI –ZnO)** in staphylococcus culture .small and non- organized shape formative around **(F- CI –ZnO)** in E coli ambient .after washing 5 times small and non- organized shape of zoon formative around both and covered large area in staphylococcus ambient than Ecoli ambient.

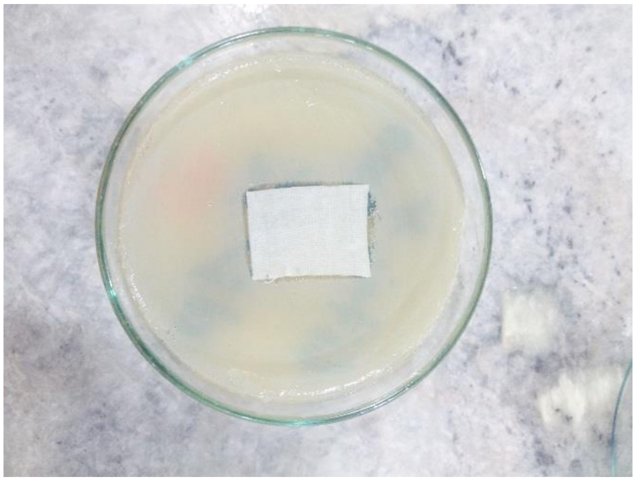


Fig (4.18) :(CI-ZnO) treated fabric with St- before washing

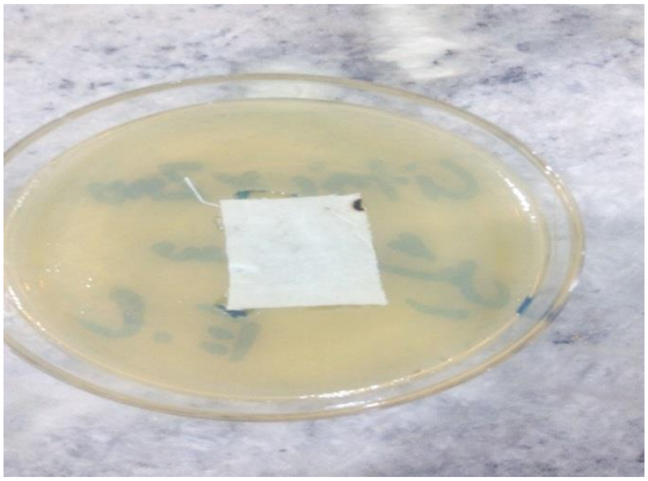


Fig (4.19) :(CI-ZnO) treated fabric with E coli before washing

Fig (4.20): (F-CI-ZnO) treated fabric with St- after washing

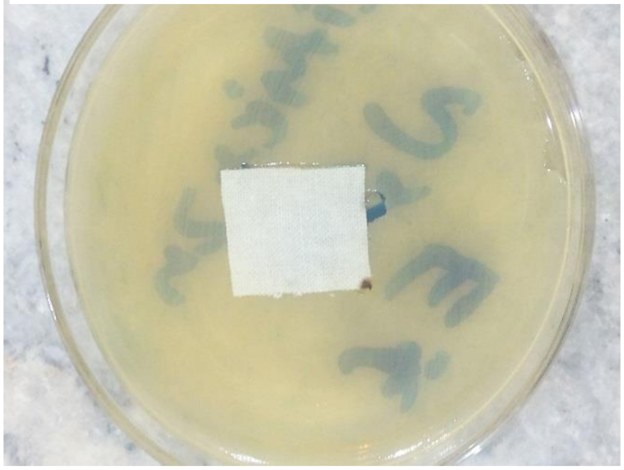


Fig (4.21): (CI-ZnO) treated fabric with Ecoli after washing

Synthesis of nano particles obtained in alkali amid (pH above 12) From anti-bacterial test it seem staphylococcus is weaker in alkali medium and E coli is weaker in acidic amid . which was obvious clearly from zoon formative around the test specimen it was larger in test before washing than after washing .Which was make alkaline specimen before washing and neutral after washing. And these led to interpretation of zoon formative before and after washing specimen due to present of ZnO nanoparticles and its bactericidal activity. This indicates that the attachment of ZnO to the cotton fabric was enhanced due to the effect of cross linking agents (Ristić et al., 2011).

4.5 Physical tests:-

4.5.1 Crease recovery:-

The cotton fiber is relatively inelastic due to it is crystalline polymer system so it is easily wrinkle and readily crease thus the blank sample shown the lowest crease recovery angle. After treatment, ZnO nanoparticles easily penetrate into the fabric pores and adhere tightly on the surface so it is not easily wrinkle or crease after treatment. on this basis cotton sample does not shrinkage when treated with ZnO nanoparticles (Shady et al., 2012)

Table (4.1): (F-ZnO)

F-ZnO	**Sample number**	**Face side**	**Back side**	**Average face side**	**Average back side**	**Total average**
Warp	1	95^0 105^0	80^0 100^0	95^0	89^0	92°
	2	80^0 100^0	85^0 90^0			
Weft	1	100^0 105^0	90^0 95^0	95^0	88^0	91^0
	2	85^0 90^0	80^0 85^0			

Table (4.2) :(F-CI-ZnO)

F – CI-ZnO	**Sample number**	**Face side**	**Back side**	**Average face side**	**Average back side**	**Total average**
Warp	1	100° 105°	80° 85°	96°	88°	92°
	2	85° 95°	85° 90°			
Weft	1	87° 100°	80° 85°	92°	86°	89°
	2	85° 95°	84° 95°			

Table (4.3): (F-A-ZnO)

F –A-ZnO	Sample number	Face side	Back side	Average face side	Average back side	Total average
Warp	1	95° 84°	85° 100°	90°	95°	93°
	2	80 90	100 95			
Weft	1	80° 85°	90° 90°	84°	93°	89°
	2	85° 85°	95° 95°			

Table (4.4): CONTROL

Cotton fabric (control)	Sample number	Face side	Back side	Average face side	Average back side	Total average
warp	1	84° 85°	90° 95°	87°	94°	91°
	2	90° 90°	95° 94°			
weft	1	90° 80°	85° 85°	86°	90°	88°
	2	80° 95°	90° 100°			

From tables 1, 2, 3 and 4, it could be observed that the crease recovery in modified fabric was increased by (1 degree) when compared to control fabric. This is mean a modified fabric will keep the good appearance for wearer to the long time (eliminated iron time). The warp CRA was greater than the weft CRA, this could be attributed to the higher number of ends than picks found in each of the case.

4.5.2 Shrinkage

The result of this test showed the dimension of warp and weft to modified fabric, still stable in the same positions before and after immersed in water, and this indicates to a good dimensions stability in modified fabrics than gray fabric.

Fig (4.8) number (1) present fabric without treated, (2) fabric modified with ZnO nanoparticle and Alovera, (3) fabric modified with ZnO nanoparticles, (4) fabric modified with ZnO nanoparticle and citric.

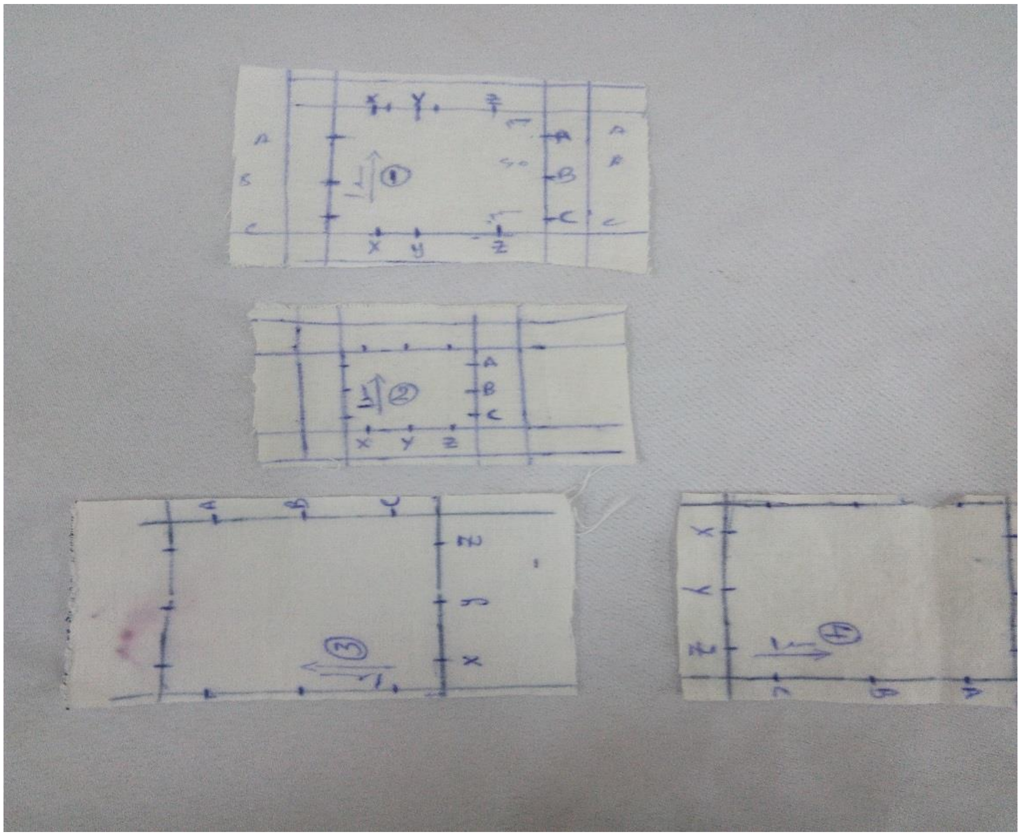
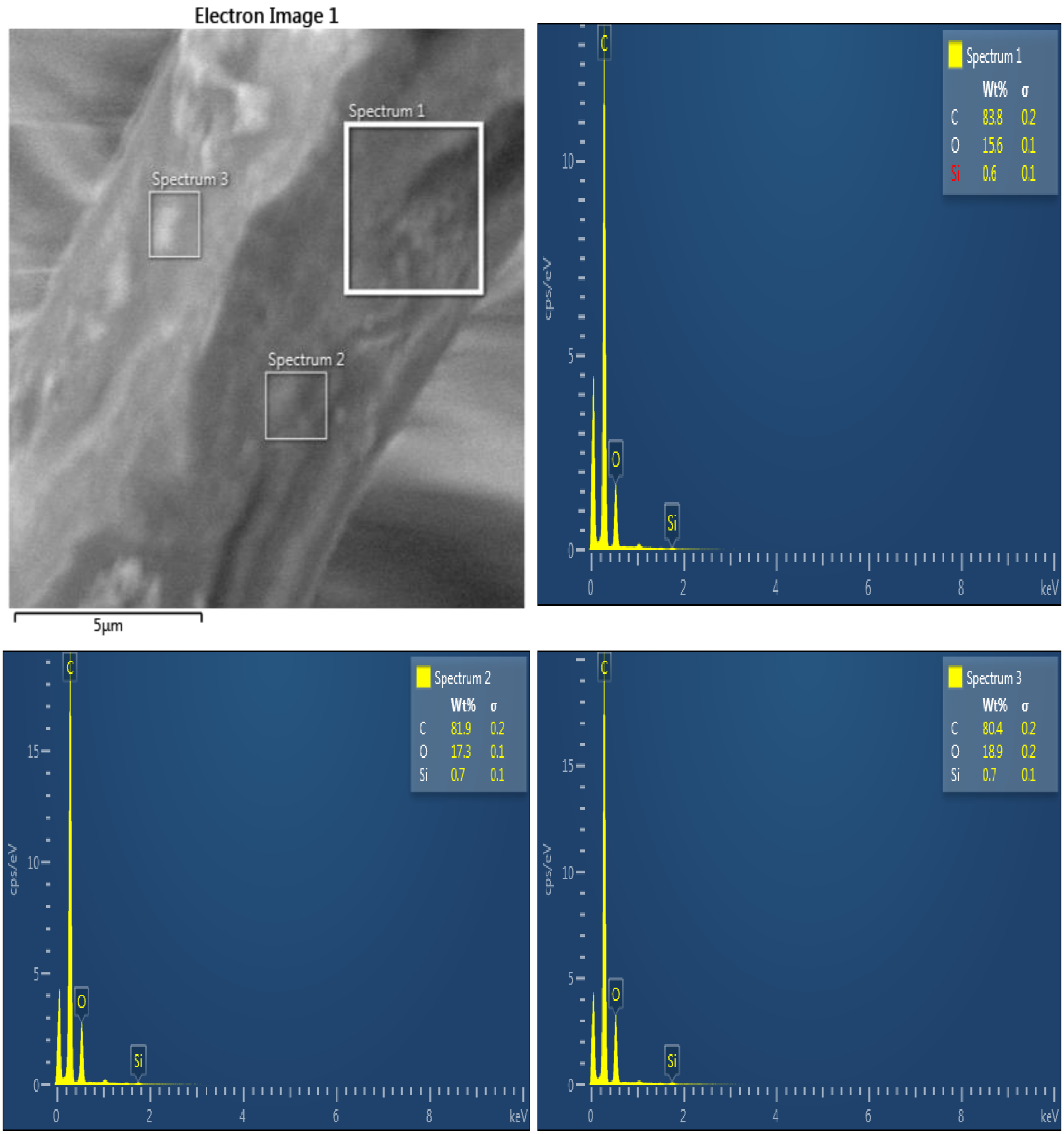


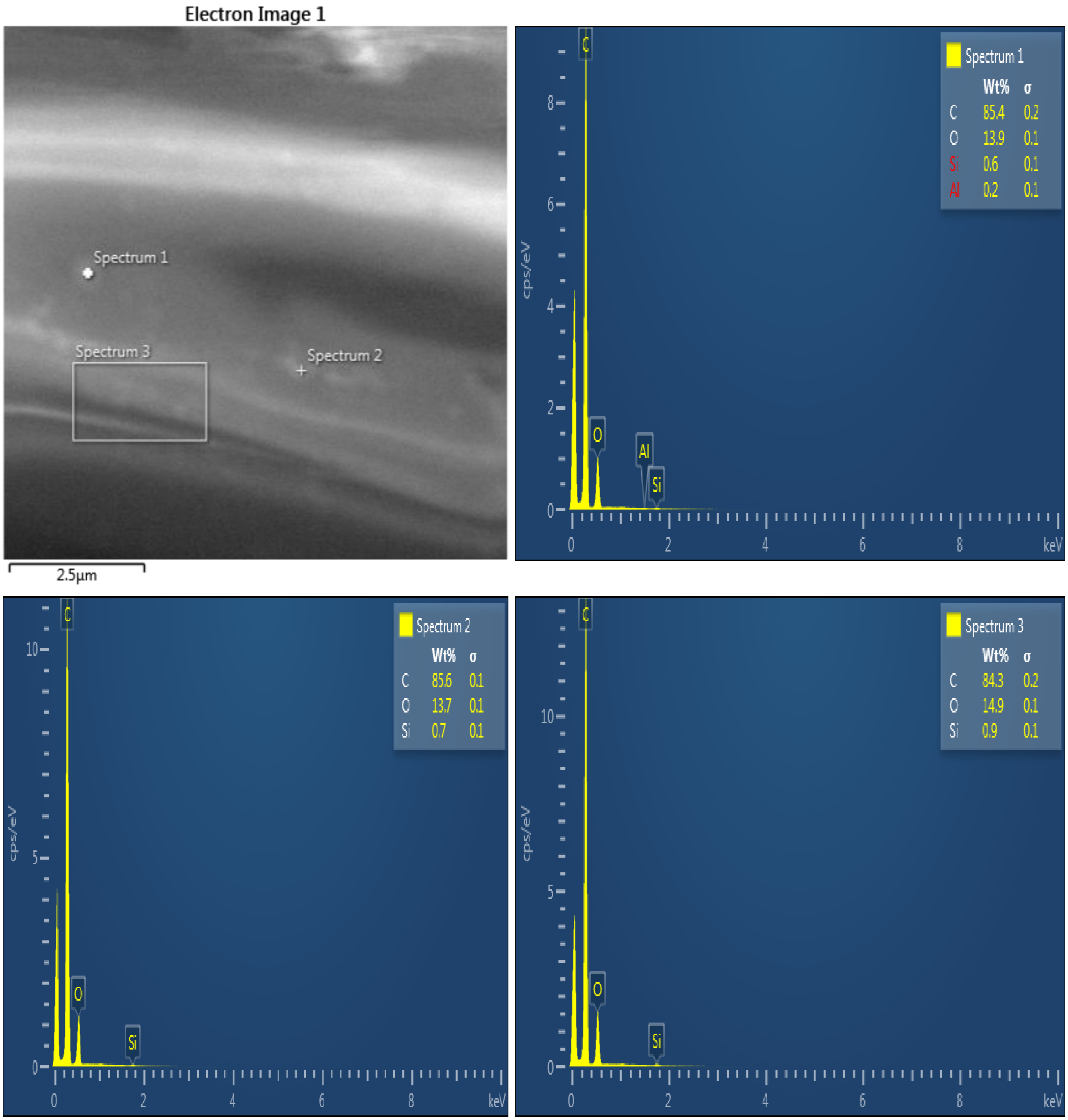
Fig (4.22): shrinkage test

4.5.3 Abrasion

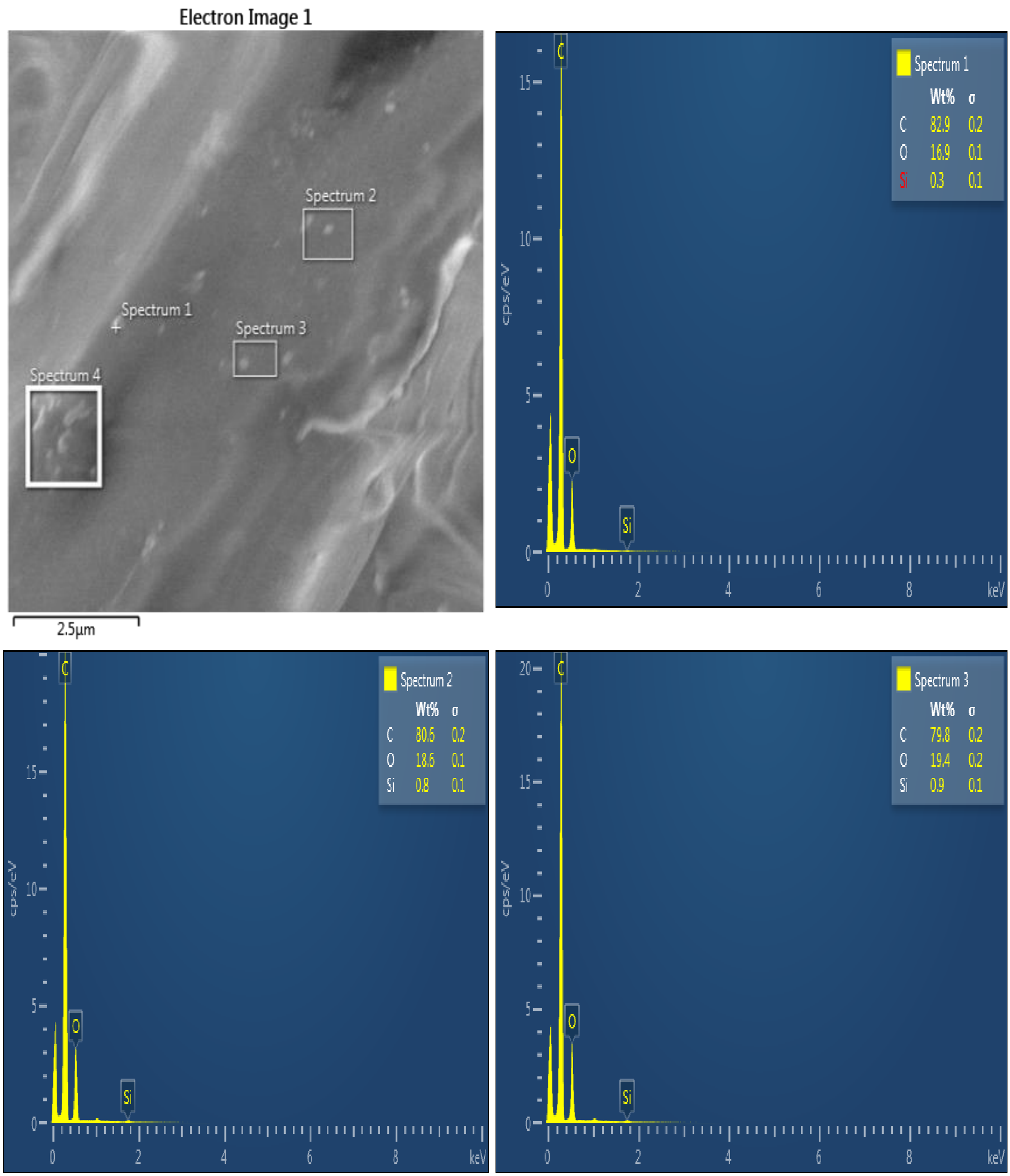
Crokometer is used to investigate the abrasion of the treated fabric. The samples (F-ZnO),(F-A-ZnO), and(F-CI-ZnO) were rubbed for 15 second instead of 10 second (ISO and ASTM) to insure of stability limit of ZnO nanoparticles on surface of fabric. Two samples of fabric were tested via crokometer instrument to study abrasion behavior, one on top(treated sample) and second on the bottom (untreated sample) , After rubbing the sample at the bottom were checked under SEM and EDS to investigate if there is any nanoparticles were released of the top sample and stable on bottom fabric surface. The result showed there is no ZnO nanoparticles removed for treated samples. The result of EDS bellow showed that.



The treated fabric with ZnO after rubbing



The treated fabric with ZnO and Alovera after rubbing



The treated fabric with ZnO and citric after rubbing

4.5.4 Tensile test

Belistone instrument was used to study tensile strength of modified and unmodified fabric, the data depicted in table (4.5) showed increase in force and decrease in elongation of modified fabric in case of Alovera and citric acid, comparing with untreated fabric. This indicate that the tensile properties of fabric has been enhanced after modification.

Table (4.5): tensile test result

samples	Elongation (mm)		Breaking Force(kg f)	
	warp	weft	warp	weft
Unmodified fabric	2.5 mm	3.7 mm	44 kg f	37.8 kg f
The modified fabric with Alovera	2.1 mm	3.5 mm	55.1 kg f	45.7 kg f
The modified fabric with citric acid	1.7 mm	3.1	50.3 kg f	39.4 kg f

CHAPTER FIVE

CONCOLOUTION AND RECOMMENDED

5.1 CONCOLOUTION:

Nano finishing is one of the most promising candidate, which is increasing significantly now a day. This Nano finishing textiles range from the protective clothing, smart textiles. Hygiene textiles, antiballistic or bulletproof vest or functionally finished clothing like water repellent or wrinkle resistance clothing.

During abrasion, laundry or thermal treatment, some of nanoparticles it could be released and reached to environments and generates environmental hazard e.g: nanoparticles may be reach inside biomolecules and cross the human cell membranes and causes cell damages**.** To increase the stability of nanoparticles and stop release of it of surface fabric natural crosslinking agents were used in this study, to synthesize ZnO nanoparticles by solution method. In this study the results showed good stability of ZnO Nanoparticles when alovera gel and citric acid were used as cross-linking agent (natural capping agent). From characterization of ZnO nanoparticle when alovera gel used as crosslinking agent the SEM, results were showed homogeneous and non–homogenous distribution of nanoparticles. However, (SEM) results when citric acid was use as crosslinking agent, exhibited homogeneous distribution of nanoparticles.

EDS results confirmed presence of ZnO nanoparticle in chemical composition of treated samples and this confirmed of successfully synthesis of nanoparticles. Also FTIR results verified the (EDS) results.

The physical tests showed the different properties of treated samples when compared with untreated sample. Crease recovery test (CRA) showed that the recovery increased in modified fabric by (1 degree) than gray fabric.

Shrinkage test indicated that a good dimensions stability in modified fabrics than gray one.

Finally, abrasion test was a doubted no ZnO Nano particles removed from treated samples.

The innovation of this study natural extract as crosslinking agent it was showed good results for all tests, from this results natural extract preferable than chemicals crosslinking agent in textile finishing.

Recommendation:

Dependent of results in this study recommended to:

- Establishment of some compounds from nature that could been used to formulate new and more potent antimicrobial drugs.
- Achieved nanoparticles stability by using natural crosslinking instead of chemicals.
- Used natural extract to enhance textile physical properties, product smart, and safety textile by applied nanofinishing technology.

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