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ORIGINAL ARTICLE

PREPARATION, CHARACTERIZATION AND EVALUATION OF BIOCOMPOSITE FILMS CONTAINING ALOE VERA AND SAGO STARCH IMPREGNATED WITH ZINC OXIDE NANO PARTICLES

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ABSTRACT

In modern science, Nanotechnology is a rage field for the researchers. Nanoparticles are used immensely due to its small size, orientation, physical properties, which are reportedly shown to change the performance of any other material which is in contact with these tiny particles and having a size of 1-100 nm in one dimension. The positive attributes of excellent biocompatibility and biodegradability of biopolymers with versatile biological activities have provided ample opportunities for further development of functional biomaterials of high potential in various fields. The biopolymers used in this study, i.e. aloe vera and sago starch are abundantly available in nature and can be used in various biomedical applications. In the present study, the composite films of aloe vera (AL) and sago starch (SG) impregnated with zinc nanoparticles (ZnNP) with and without antibiotic gentamicin (G) were prepared by solvent casting method. The films prepared were characterized for their physico-chemical properties using conventional methods like tensile property, Water absorption studies, Fourier transform infrared spectroscopy. From the physicochemical results, it was proved that the addition of Aloe vera increases the tensile strength and water absorption capacity of the SG film. But when the zinc oxide nanoparticles were added to SG-AL composite film, it showed a slight decrease in the water absorption capacity. The films prepared were characterized by FTIR and found that it is composite in nature. The SEM picture showed the porous and rough surface of the film which is one of the main characteristic of wound material. The UV visible absorption spectra of the absorption of pure ZnO nanoparticles exhibit the peak at 345 nm. This study suggests that Av-SG-nZnO film may be a potential candidate as a dressing material for wound healing applications.

Keywords: Sago starch, *Aloe vera*,(AL) Biocomposite , FTIR, EDAX,SEM and Zinc oxide nano particles

1.INTRODUCTION

Nanotechnology deals with production, manipulation and use of material ranging in nanometres (Kavitha *et al.*, 2013). Human life gets an impact role in all sphere mainly in the field of nanotechnology (Jannathul Firdhouse *et al.*, 2012). The field of nanotechnology is one of the most active areas of research in modern material sciences. Nanoparticles exhibit completely new or improved properties based on specific characteristics such as size, distribution and morphology. Nanoparticles of noble metals, such as gold, silver, platinum and zinc oxide are widely applied in products that directly come in contact with the human body, such as detergent, cosmetic products and tooth paste, besides medical and pharmaceutical applications.

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Nanotechnology is a potentially beneficial field with tremendous implications for society, industry and medicine. The uses of nano-sized particles are even more remarkable (Siracusa *et al.*, 2008). They are mostly prepared from noble metals like Gold, Silver, Platinum, Zinc and Palladium (Jagannath, *et al.*, 2006). They find applications in various fields like medicine, electronics, textile, cosmetics, and so forth. The plant mediated synthesis is rapid, low cost, eco-friendly and for safer human therapeutic uses (Darder *et al.*, 2007). In general, natural polymers can be polysaccharide- or protein- based. Starches are universally available with low cost and good film formability. Among the starch materials, sago starch is relatively unknown. It is obtained from an uncommon source (Metroxylonsagu palm tree) in Southeast Asia at a very low cost compared with common starches. For the past decade, nano scale science and other related technologies has been the leading technology (Narayanamurti, 2006). Incorporation of nanoparticles into composite materials has attracted a great deal of attention due to its ability to enhance polymer properties such as thermal, mechanical, and gas barrier (Kurian *et al.*, 2006). In addition, the use of zinc oxide nanoparticles is considered to be a viable method for the prevention of infectious diseases through the antimicrobial effects of zinc oxide (Li *et al.*, 2009 and Zhang et al., 2010). The size, morphology, crystallinity, composition, and shape of particles are critical parameters of the intrinsic properties of nanoparticles (Shahrom, and Abdullah, 2007).

Nano materials have attracted tremendous interest due to their noticeable performance in electronics, optics, and photonics. Nano materials are typically classified into three groups: 0- dimensional, 1-dimensional and 2-dimensional. 0-dimensional nanostructures, referred to as quantum dots or nanoparticles with an aspect ratio near unity, have been extensively used in biological applications (Liu *et al.*, 2010 and Hoshino *et al.*, 2004).

In addition to the skin, other epitheliums in our body include the lining of the gut, the bronchial tubes and the genital tract, which also benefit from the healing effect of A. vera (Joseph and Raj, 2010) When taken internally, A. vera juice aids the digestion and absorption of nutrients, helps control blood sugar, increases energy production, promotes cardiovascular health, improves liver function, and boosts the immune system.Thepulpis used extensively for treating constipation, enlargement of spleen, zymotic disease and chengamaari (a type of venereal infection). For balancing digestion and elimination,take1table spoon A. vera gel in the morning on an empty stomach. Aloe helps clear the toxins out of the digestive system, facilitates digestion and improves the functioning of the kidneys, liver and gall bladder (Agarwal and Sharma, 2011).

In this study, a novel low cost composite film sago starch with aloe vera gel was prepared and it was impregnated with zinc nanoparticles. These prepared films were tested for physicochemical methods.

2. MATERIALS AND METHODS

MATERIALS

The materials used for this study include sago starch which was purchased from nearby local retail market and other chemicals used in this study were purchased from Merck Chemical Co.

METHODS

Plant collection and preparation of aloe vera gel (AL) 2.2.

Fresh leaves of *Aloe vera* were collected from Kumarapettai village, Cuddalore district, Tamilnadu, India. The leaves of *aloe vera* was washed with distilled water and outer layer of the leaf was peeled using knife and inner portion was collected. The collected samples were crushed by using electronic mixer and filtered. Then the filtered solution was stabilized at 65^{o}C for 15 minutes and stored at 4 ^{o}C for further use.

Preparation of zinc oxide nanoparticles (nZnO)

Zinc oxide nanoparticles prepared by sol-gel method of *S.Nayar et al.* 40 mg of sodium hydroxide in 100 ml of methanol was added dropwise to 0.1 M zinc acetate and the solution was mixed continuously for about 20 minutes. After 10 minutes of mixing, zinc oxide nanoparticles were obtained as a white colloidal dispersion in methanol and the resultant solution were centrifuged to remove unreacted precursors.

Preparation of Sago starch (SG) solution

Sago was powdered using a domestic mixer and sieved to a particle size of 50–200 µm. 10 g of sago powder was boiled in 100 ml water till it becomes a homogenous solution. The solid content of the solution was found to be 13.88% and this solution was used for further experiments.

Optimization of sago starch (SG) films

As sago starch film was brittle, ethylene glycol was used as a plasticizer to enhance the film flexibility. Keeping the amount of sago starch solution constant, the amount of ethylene glycol added was varied and the resultant solution was poured into polythene trays having measurements 10x10 cm and dried at 30-35^{o}C. The dried films were stored in polythene cover for further use.

Optimization of Sago starch – Aloe vera (SG-AL) films

The extracted aloe vera gel in the ratio 5%, 10%, 15%, and 20% was added to the SG solution having the better tensile strength. The blended solution was homogenized using magnetic stirrer at room temperature for 20-30 minutes and the resultant solution was poured into polythene trays having measurements 10x10 cm and dried at 30-35^{o}C. The dried films were stored in polythene cover for further use.

Table:1. Mechanical properties of SG film

Sample number	Sago (ml)	Ethylene glycol(ml)	Elongation at break (%)	Tensile strength MPa
1	40	1	__	__
2	40	1.5	35.23 ± 2.76	3.1 ± 0.29
3	40	2.0	32.06 ± 0.58	9.21 ± 0.68
4	40	2.5	36.68 ± 3.32	2.39 ± 0.28

Optimization of SG-AL -nZnO films

The stoichiometric ratio which gave better tensile strength to SG-AL film was used for the preparation of SG-AL- nZnO composite film. To the SG-AL film, 1% of freshly prepared zinc oxide nanoparticles was added and stirred continuously for 15-30 minutes and the resultant solution was poured into polythene trays having measurements 10x10 cm and dried at 30-35^{o}C. The dried films were stored in polythene cover for further use.

Table 2: Mechanical properties of SG-AL film

Sample number	Sago (ml)	Ethylene glycol(ml)	Aloe gel (ml)	Elongation at break (%)	Tensile strength MPa
1	40	2	5	27.89 ± 4.75	5.5 ± 1.35
2	40	2	10	35.49 ± 0.58	11.08 ± 0.78
3	40	2	15	33.68 ± 2.34	8.20 ± 1.72
4	40	2	20	24.42 ± 3.72	6.15 ± 0.89

Sterilization of the films

The prepared films were stored in polythene covers and the films were sterilized for 4 hours at 45^{o}C with 100% ethylene oxide (Eto) and stored for further use.

Characterization

Tensile strength

Three dumbbell-shaped specimens of 4 mm wide and 10 mm length were punched out from the prepared films using a die. Mechanical properties such as tensile strength (MPa) and percentage of elongation at break (%) were measured using a universal testing machine (Instron model 4501). The break load was measured and the tensile strength was calculated by the following equation

Tensile strength = Break load/Strip cross-sectional area

Water absorption capacity of the samples

Water absorption capacity was done by the method of Rao et al. The samples of known weight were swelled in distilled water at room temperature. The swollen weights of the samples were determined by first blotting the samples with filter paper followed by accurately weighing the sample. The weights of the swollen pieces were recorded every 1 h, 2 h, 3 h and after 24 h. Percentage swelling of the samples at a given time was calculated from the formula

$$WS(\%) = [(W_s - W_o) / W_o] \times 100$$

where Ws is the weight of the sample (moist) at given time, Wo is the initial weight of the sample, Es is the percent of swelling at a given time.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra of the samples prepared were taken using Nicollet impact 400 FT-IR spectroscopy by preparing a 500 mg KBr pellet containing 2–6 mg of the sample.

UV-Visible Spectroscopy Analysis

Zinc oxide nanoparticles were characterized in a Perkin-Elmer UV-Visible spectrophotometer. The reduction of pure Zn+ ions was monitored by measuring the UV-Visible spectrum by diluting a small aliquot of the sample into distilled water. UV-Visible spectral analysis was done by using UV- Visible spectrophotometer at the range of 300-450 nm and observed the absorption peaks at 325-345 nm regions, which are identical to the characteristics UV-visible spectrum of Zinc oxide and it was recorded. The spectrophotometer was equipped with "UV Winlab" software to record and analyze data. Base line correction of the spectrophotometer was carried out by using a blank reference.

Statistical analysis

Data are expressed as means + SD. Analysis of variance (ANOVA) followed by the student's t-test was used to determine the significant differences among the groups. P-values less than 0.05 were considered significant.

3.RESULTS AND DISCUSSION

Table 3. Description of the films

Sl. No	Types	Description
1.	Control	Sago starch films (SG)
2.	Type – I	Sago starch incorporated with Aloe vera extract(SG-AL)
3.	Type –III	Sago starch and Aloe vera extract incorporated with Zinc oxide nanoparticles (SG-AL -nZnO)

Table 4. Water absorption studies of the biocomposite film containing SG,SG-AL, & SG-AL-nZnO films.

HOURS	(SG) (%)	(SG-AL) (%)	(SG-AL -nZnO) (%)
1hr	38.49 ± 5.8	63.34 ± 6.58	58.23 ± 2.78
2hrs	40.37 ± 4.36	73.70 ± 7.33	65.08 ± 4.31
3hrs	42.37 ± 6.73	75.90 ± 5.11	68.76 ± 6.13
24hrs	50.32 ± 3.89	76.50 ± 4.89	75.11 ± 3.23

Values are given as mean ± standard deviation. (p<0.05)

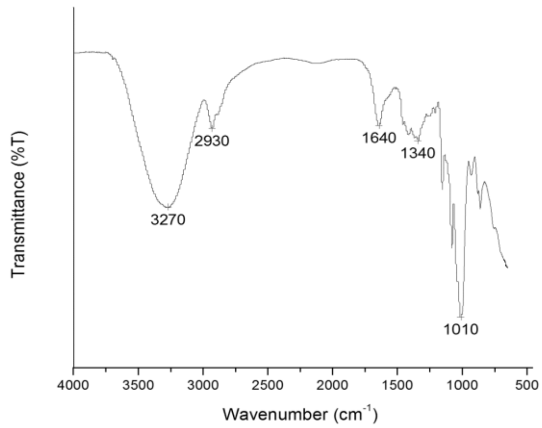

Fig 1. FTIR spectra of SG film

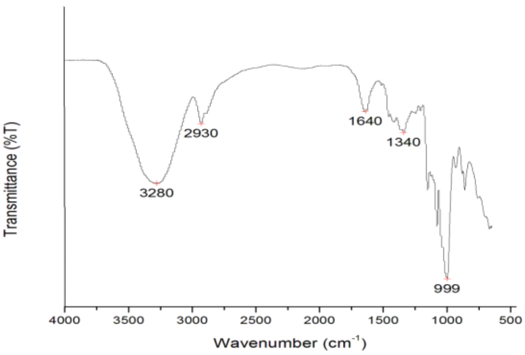

Fig 2. FTIR spectra of SG-AL film

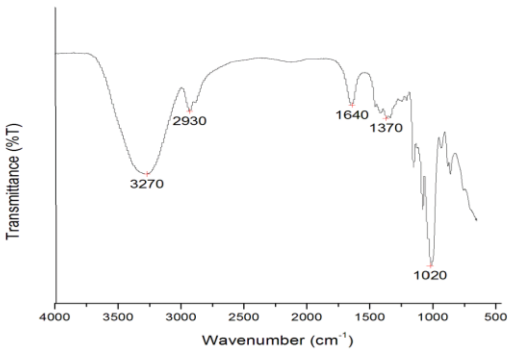

Fig3. FTIR spectra of SG-AL –nZno film

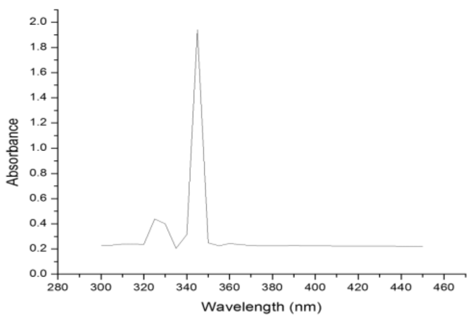

Fig 4.UV spectra of ZnO nanoparticles

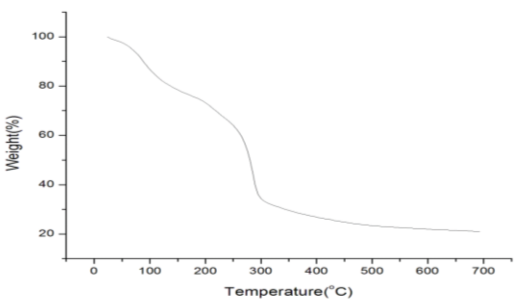

Fig 5. Thermogram of SG-AL -nZnO film

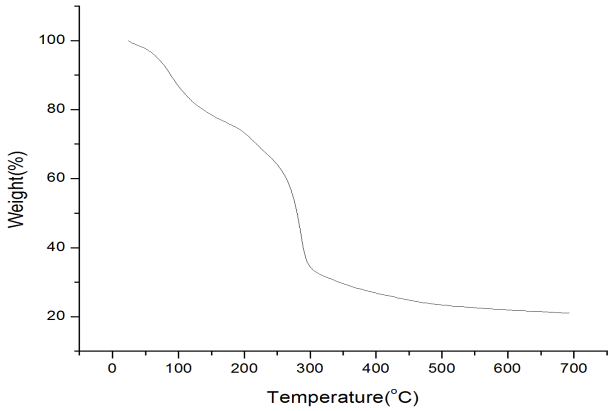

Fig 6. Thermogram of SG-AL film

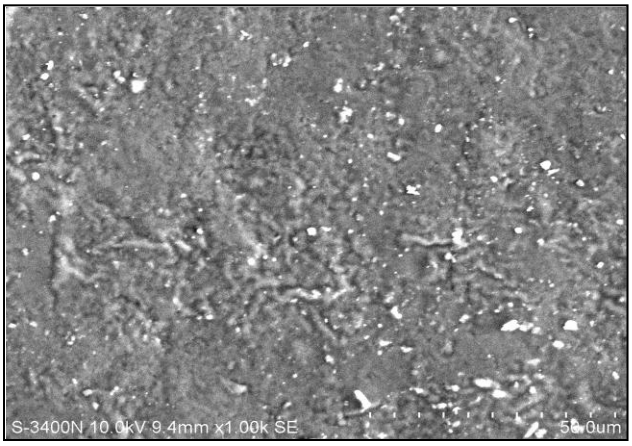

Fig 7.SEM micrpgraph of SG film

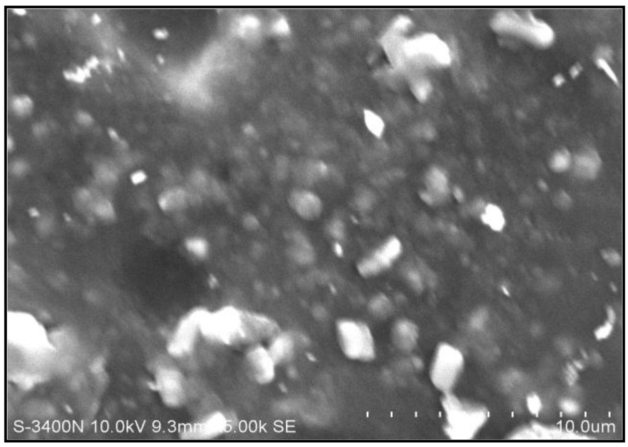

Fig 8. SEM micrograph of SG-AL film

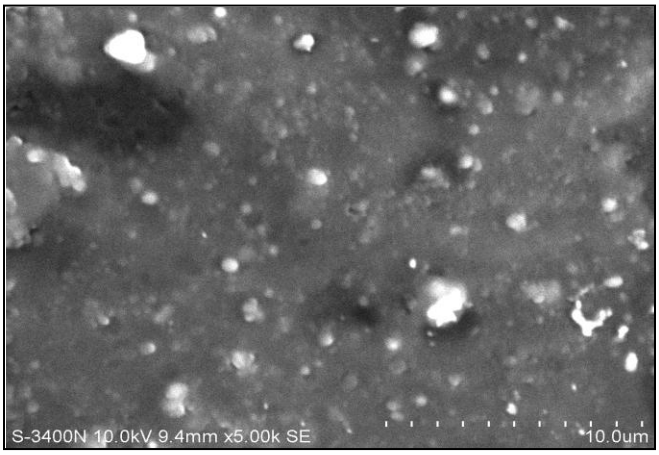

Fig 9. SEM micrograph of SG-AL film with ZnO nanopaticles

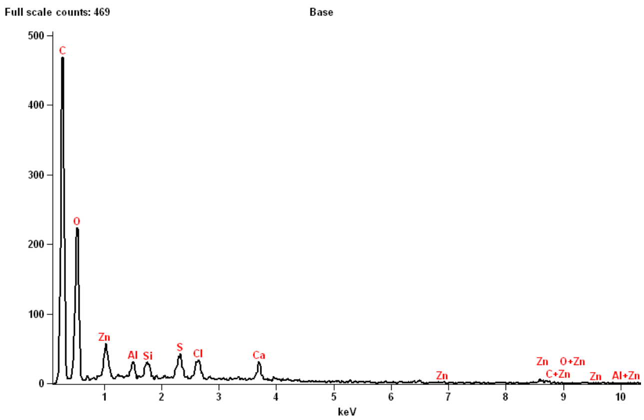

Fig 10. EDAX spectrum of SG-AL -ZnO film which shows the presence of Zinc.

Tensile property

A wound dressing material should have a good mechanical property in order to be used as good wound dressing material. To increase the mechanical property, SG film was incorporated with Aloe vera in different ratio as shown in the table 2. The SG-AL film which gave good tensile strength was used to prepare SG-AL the film with better mechanical property was taken to form SG-AL -nZnO film.

Water absorption property

Water absorption capacity is an important property for a wound dressing material as it absorbs wound exudates and keep the wound surface dry and thereby enhancing the wound healing. Keeping this in view, water absorption studies of the samples prepared were studied. The water absorption capacities of samples SG, SG-AL, and SG-AL-ZnO were SG-AL film has shown increased water absorption capacity with increase in time, a maximum of 75 % water absorbing capacity was absorbed after 24 hrs.

SG-AL film has also shown increased water absorption with increase in time as that of SG-AL film water absorption capacity was higher than SG film.

Thermogravimetric analysis

The initial rate loss (below 100^{0}C) observed in the films is due to loss of moisture present in the films. The weight loss observed in the case of SG film is 70 % at 320 ^{o} C . whereas SG-AL film at this temperature 71.33%. The weight loss difference between SG-AL and SG-AL and -nZnO films represent the presence of zinc oxide nanoparticles in the SG-AL, nZnO film. The 0.5 % amount of zinc oxide nanoparticles present in the SG-AL -nZnO film can be calculated from the weight loss difference, SG-AL and SG-AL and nZnO films at 700^{0}C was 0.1 %. At this 700^{0}C, the thermogram of SG, SG-AL, and SG-Al and -nZnO film has shown 18%, 16%, 12% and 20% residual matter respectively. The films samples of SG-AL and SG-AL -nZnO have not shown a significant weight loss difference in thermogravimetric analysis.

FOURIER TRANSFORM INFRARED SPECTROSCOPY

The FTIR spectra of SG, SG-AL, and SG-AL -nZnO films were shown in the figures 1,,2 and 3. respectively. The FTIR spectra of SG showed typical absorption bands of a starch backbone. The absorption bands at 3291 cm^{-1} are due to O-H stretching vibration. Usually, the absorption range for O-H vibration is 3700-2500 per cm^{-1}, but in sago starch the O-H stretching peak shifts to lower wavelength. This is probably due to the intermolecular hydrogen bonds in the glycosidic ring which weakens the O-H bonds, thereby shifting the band absorption region to lower frequency between 3400-3200 per cm^{-1}.The absorption band at 2929 cm-^{1} shows the C-H stretching. The weak absorption at 1627 cm^{-1} for sago starch probably due to the tightly bound water molecules in the starch.

The ⁻CH_2 symmetrical band is found at 1335 cm^{-1}. The broad absorption band in the range of 1100 to 990 cm^{-1} characteristics of C-O stretching in C-O-C and C-O-H in the glycosidic ring of sago starch. In addition, broad and weak bands occurred in the range of 930 to 600 per cm-1, probably arising from out of plane bonded O-H deformation and C-H deformation frequencies. The absorption bands of COO- groups were seen at around 1643 cm^{-1} (asymmetric COO- stretching vibration).

UV SPECTRA

The UV visible absorption spectra of prepared nano ZnO. In this UV spectra, the absorption of pure ZnO exhibits UV absorption at 345 nm. This can be referred to as the exciton absorption peak of ZnO nanospears. The excitonic peak at 345 nm and 325 nm showed that the particles formed was that of low range. The sharp peaks further indicates that the synthesized particles were rather monodispersed.

SCANNING ELECTRON MICROSCOPY (SEM) AND ENERGY DISPERSIVE X-RAY ANALYSIS (EDAX)

Marie Arockianathan *et al.*, (2012) prepared the composite films of alginate (AL) and sago starch (SG) impregnated with silver nano particles (AgNP) with and without antibiotic gentamicin (G) by solvent casting method. The films prepared were characterized for thermo gravimetric analysis, SEM, TEM and mechanical properties and the results have shown the composite nature of the films.Mariam *et al.*, 2014 reported a novel synthesis for In2O3 and ZnO Nanoparticles with particle sizes in the range of 10 to 30 nm using indium nitrate and zinc nitrate solutions. They utilized A. vera extract as a solvent instead of organic solvents. The EDAX shows characteristic Zinc oxide nanoparticles peak.

Sarah Ibrahim hashoosh *et al.*, (2014), explained the role of *Aloe vera* extract as a reducing agent for the production of Ag nanoparticles. The UV-VIS spectrophotometer showed shift peak at 400nm and scanning electron microscope (SEM) showed the rectangular morphology of as prepared Ag nanoparticles with a size of 500 nm.

Scanning electron microscope pictures exhibit the surface morphology of biomaterials. In the present study, scanning electron microscope pictures were taken for SG, SG-AL, and SG-AL -nZnO films (Figures 7,8,& 9). The SG film has shown porous and rough surface, similar surface characteristics were observed for SG-AL, and SG-AL and nZnO films. The porous nature of the SG-AL -nZnO film helps in absorbing the wound exudates and also helps in oxygen exchange to the wound surface. The EDX spectrum of SG-AL -nZnO film (Figure 4) has clearly shown the presence of Zinc with its corresponding signal peak.

4.CONCLUSION

In the present study, the novel biocomposite films containing sago starch/ turmeric/ aloe vera impregnated with zinc oxide nanoparticles were prepared, characterized and evaluated. Since zinc is one of the essential trace elements for human body that is under strict regulation, bionanocomposites based on zinc oxide nanorod may have potential applications in the medical, pharmaceutical, and food packaging industries. This showed the biocompatible nature of the material and it did not show any cytotoxicity. By these results, it was clear that this material might act as good wound dressing material in future and it can be tested in the laboratory on smaller and bigger animals to prove its efficacy. The method was unique, cost effective to biosynthesized nanoparticles and other nanocomposites from the natural resources. Still more clinical trials need to be conducted to support its therapeutic used.

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