Fabrication and Optimization of Low Emissivity Polymeric films for Energy Efficiency

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Dedication

"I dedicate this thesis to my loving and supportive Father and my beloved Mother and teachers."

Acknowledgments

All admiration to Allah Almighty who is the creator, maintainer, and the Regulator of the world. He is the One, who bestows and gives the power to us to think, utilize our expertise in knowledge in achieving remarkable solutions for mankind in every field. Therefore, I express my greatest thanks to Almighty Allah the universal and the architect of the world, who has gifted us the brain and instable nature construction of knowledge and body to achieve our work in the form of this project report. As Allah Almighty says in Quran:

"Read! In the name of your lord" (Alaq; 1st revealed ayah)

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-Kanza Iqbal

Abstract

Energy crisis is the major problem of the modern era. In buildings cooling requirements are due to solar heat gain through windows. To overcome this problem, there are variety of films have been made by different methods and using different polymers. In this research a solution casting method used to fabricate low-emissivity window films using flexible polymeric substrate incorporated by metal dust. These window films further characterized by using Scanning Electron Microscopy, X-rays Diffraction, UV-Vis-NIR Spectroscopy, Heat radiometer test , Contact angle and Mechanical testing to examine the morphology, phase, absorption, transmittance, reflectance, heat flow, hydrophobic/hydrophilic nature and mechanical strength . Fabricated films show emissivity values of 3.2 W/m^2 K to 3.3 $W/m^{2.}$ K for Al/CA and 3.1 W/m^2 K to 3.8 W/m^2 K for Sn/CA whereas solar radiations reflectance about 80% in infrared region. The characterized low emissivity films incorporated by metal dust are tinted, uniform, and flexible hydrophilic films exhibits excellent mechanical performance for window application.

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List Of Abbreviations

Low E…………………………………………………...Low Emissivity

SHGC……………………………………...………...Solar Heat Gain Coefficient

VT…………………………………………………...Visible Transmittance

IR…………………………………………………… Infra-Red

NIR…………………………………………………...Near infrared

Chapter 1

Introduction

Nanotechnology is a fast-growing field entering the world of smart materials and taking them to the next level. Nanotechnology is a promising field; it promises vital improvements in manufacturing techniques and advanced materials[1].It has applications nearly in every field of research. Nanotechnology has so much potential in research and development to address major problems. The world is facing a severe energy crisis. The need of the hour is to shift towards sustainable and green energy products. Due to the increased human population and growth in energy consumption, the world 's energy resources are depleting day by day[2] [3]. The dependence on fossil fuels like coal gas and natural oil is exceeding at an alarming rate. Due to this, fossil fuels are draining excessively. Also, the burning of these materials produces gases like CO_2, SO_X, and NO_X which are not only causing pollution in the environment but are also a hazard for life on Earth due to global warming and the greenhouse effect.

The increasing usage of glass and plastic in Building exteriors, auto-industry, indoor household applications, solar panels, etc. have made its cleaning an additional step to the maintenance procedure. A considerable amount of money is spent per annum in cleaning exteriors of high-rise buildings and solar panels, the former being a necessity in areas that are prone to dust and the latter being essential for efficient working of the solar cells. Therefore, the growth of self-cleaning materials and the link between structure-function associations is an active research area in Nano science technology. The fact that type of materials can be easily cleaned even from a stream of rainfall suggestively diminishes the routine maintenance price [4].

Over the years, the utilization of glass for both household and commercial applications has increased exponentially. With the growing trend of high-rise buildings, one problem has become imminent- the costly maintenance (both financially and human resource-wise) of cleanliness and uncontrollable energy losses from glass applications (e.g. windows). This problem extends to other applications of glass such as solar panels as well. It is also imperative that buildings should remain cool from within in summers and warm in winter. Every year, a large fraction of expenditure goes under the banner of maintenance and expenses of air-conditioning or heating buildings centrally. Given this, we realize it’s an impending necessity to develop a self-cleaning and low emissivity glass coating. Self-cleaning coatings using thin film or nano-particle semiconductors have gained increasing interest since the last decade. [5]–[8] Due to the commercialization prospect and extensive practical applications such as window glass fabrics, paints, construction materials, plastics corrosion protection, and solar cells [8]–[10].

Emissivity is the property of a surface to emit out or radiate energy. When any surface is exposed to energy, some of it is absorbed; some is reflected while the rest is transmitted through. Every material including glass windows emits energy in form of radiation. These could be long or far infra-red radiations, depending on the temperature of the surface. Thus, better the insulating properties of a window; one must reduce the window’s emittance. The glazing market has been revolutionized with the introduction of low-E glass in the market. The need of the hour is to interpret the diverse window properties, their parameters, and advantages. The properties to be measured are such as thermal emittance, total solar energy transmittance, visual transmittance. Also, the understanding of the materials needs to be done for their employment. The glazed windows are employed in cold regions to make energy-efficient glass coatings. The thermal emittance values of uncoated glass have been reached approximately 84%,which can be reduced to 15% [11].Many materials are being exploited, which do not only provide renewable energy but are also cost-effective. The need for the hour is to prepare environmentally friendly and green products so that these energy harvesting devices do not pollute the environment. Since then, many materials have been synthesized and many devices have been fabricated like solar cells, wind energy, fuel cells, capacitors, supercapacitors, etc. [12] [13]. which not only can produce renewable energy but also can store energy for long power run [14]–[17]. The increased usage of energy and reliance on portable energy devices is demanding increased energy and power density [18], [19].

1.1 Renewable Energy Resources

Renewable energy resources are those which cut our dependence on non-renewable energy resources like fossil fuels [20] [13]. These resources do not deplete, and they can be reused again for our convenience. There are many forms of renewable energies which include:

a) Hydrothermal energy [21]
b) Geothermal energy[22]
c) Solar energy [23], [24]
d) Wind energy [25]
e) Biomass [26] [27]

Renewable energy sources should be used as they are natural and better. They are also cost-effective and green. They do not create havoc for the environment. They are getting cheaper by the day as technology is progressing. New technologies are also being explored in them.

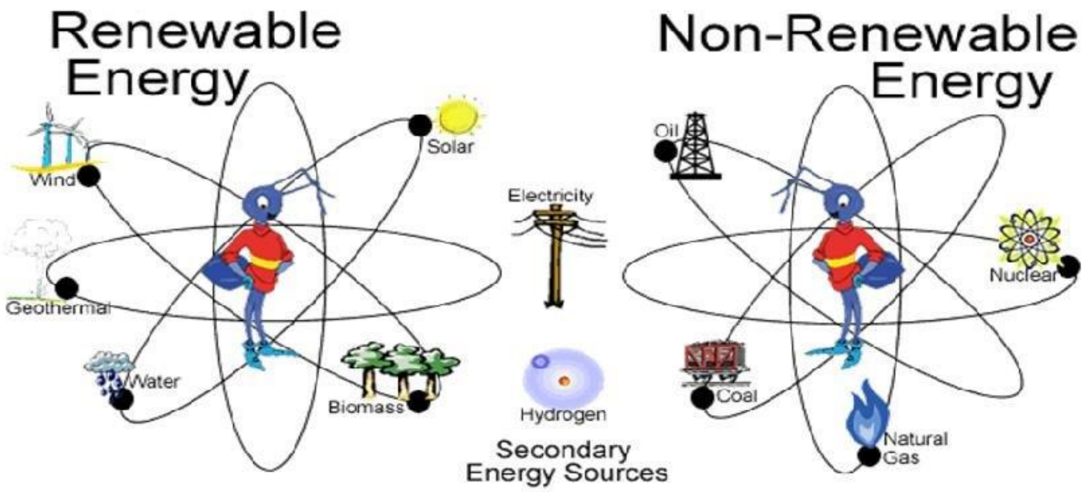


Figure 1.1 Renewable/Non-Renewable Energy[28]

1.2 Energy Management:

Out of all the energy resources, solar energy is now widely applied for our energy needs. It has enormous advantages as compared to other energy resources. As fossil fuels are depleting, the need of the hour is to shift toward renewable and green energy. Solar energy can be utilized for generating electricity, heat, and fuel which can compensate energy demands as it is cleanest and most abundant in nature. This energy is exploited on large scale to fulfill the daily demands of energy. Solar energy is more probable to meet global dependence on fossil fuels.

It is completely free and readily available. Solar energy can be harnessed with modern-day technologies like photovoltaic energy devices[29]–[31], solar heating[32], [33], and also artificial photosynthesis[34], [35]. Solar energy can be directly converted to electricity by making portable energy devices like photovoltaic devices[36]. Solar energy can be distinguished as the future energy source as it is clean and non-polluting. This means it can combat the greenhouse effect caused by the excessive use of fossil fuels. It is ecologically acceptable. It is a reliable source of energy. It is also noise pollution-free as it only requires sunlight to generate electricity and no additional maintenance is required for a long go. It can be harnessed for the degradation of organic environmental pollutants produced via industrial wastes or the burning of fossil fuels.

In trend mostly, buildings waste a large portion of the energy consumption up to 30%, due to cooling and heating loss from the building. Losses from windows itself are approximate to cost U.S. consumers approximately 40 billion dollars per year. In standard windows, 60% of the total heat loss is due to radiation loss. The question remains that how the efficiency of the windows is controlled when heat passes through the window and the most cost-effective solution is through applying the low-emissivity (low-E) films and coating. [37]–[39].

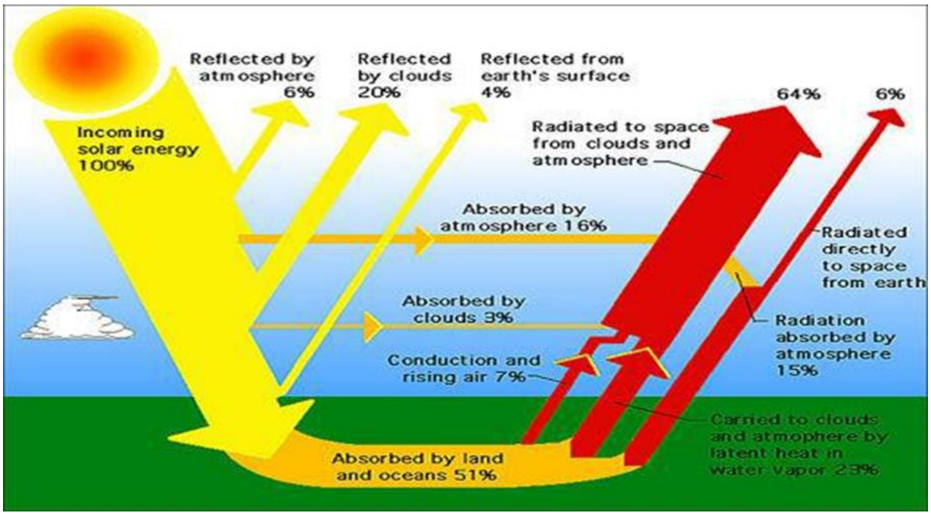


Figure 1.2 Solar energy division on earth[40]

1.3 Energy and Low Emissivity Films:

Window films have been used in many notable places for a long time, but the key point of architectural films is the ability to minimize energy costs. Modern in the window film manufacturing industry using all types of metals Colorless films to alteration color and advanced adhesive that adhere to the films. Films offer about 10 to 15 (years) of warranties for many used products and often lifetime warranties. Window films are now protected by layers called UV absorbing layers which block more than 99% UV rays and also provide scratch-resistant coating [41], [42].

Most of the window films consist of a thin-film polyester substrate that has a transparent metal coating of micro-size on one side which absorbs and reflects the radiation before it transfers into a building. Minimizing the solar heat gain (SHGC) and building cooling load from windows, window films also minimalize total time that cooling equipment of the buildings must have to keep comfortable zones which eventually results in less electricity. Savings of 5-15% in total building electricity costs, kilowatt-hour (Kwh) consumption, and kilowatt(kW) peak demand can be taken, with the savings dependent upon several factors, like

- Window to wall ratio.
- Presence of overhangs.
- Type of glass.
- Climate.

- The performance level of the film.
- The efficiency of the building cooling equipment.

Probably Low-E films having an emissivity range of about 0.33 and approximately 67% of heat is reflected during winter that drastically reduces the cost of heating and keeping energy consumptions lower in both summer and winter.

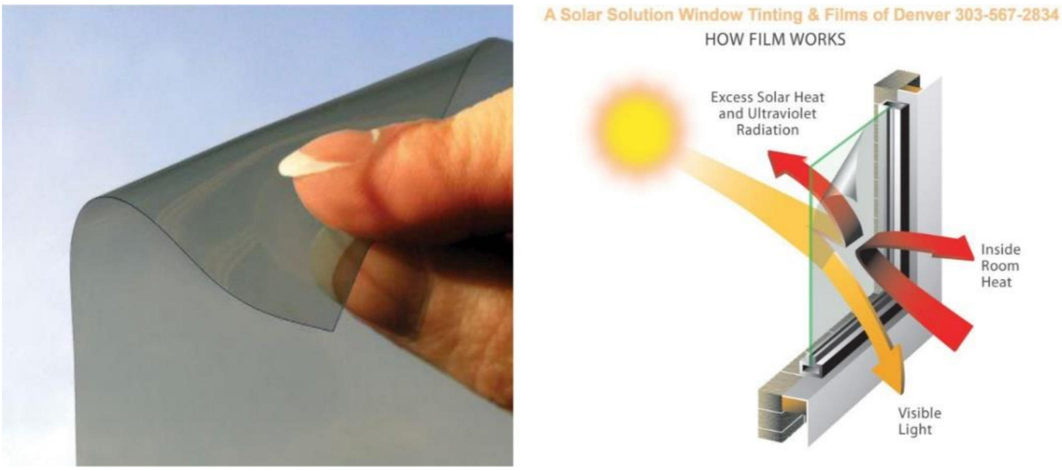


Figure 1.3 Low emissivity film with window[43]

1.4 Low-E Film Effectiveness:

There are a variety of benefits by selecting low e film for buildings, especially reduce the cost as well as energy and low heating and cooling electricity bills

Easy installation:

A low Emissivity film sticks on the glass directly so installation is fast and easy with the benefits of low e film immediately.

Good light transmission:

A low-Emissivity film permits you to reduce SHGC, however still enjoying excellent transmission of visible light meaning your home keeps bright and brings natural light.

Flexible:

Low-E films in a combined form with different glass types they can suit the design or aesthetic aspect for buildings.

Reduced electricity bills:

In winter heat loss prevention and other is in summer heat gain resistance, your electricity bills fall by using Low-E film. Report shows a good improvement in its U value, SHGC. The wide number of incentives both economically and environmentally Firstly consider your circumstances carefully Analysis of the home location or direction that it faces, check out which windows and doors more benefit from Low-E film with maximum exposure to the sun.[44]

Figure 1.4 Low-E film summer and winter effect [45]

Figure 1.5 Low Emissivity film installation[46]

1.5 Metal

Metal from any class specified by several properties like high thermal conductivity, electrical, conductivity, malleability, ductility, and high reflectivity. Almost all discovered chemical elements quarter third are metals. Abundant varieties of metals are sodium, aluminum, iron, calcium, potassium, and magnesium. Metals exist in the form ores but few of them occur as gold, copper, silver, and platinum in the free state as they are not ready to react with any other elements[47], [48].

Metals are crystalline solids that have a simple crystal structure differentiate by atomic packing fraction and highly symmetric. Metal atoms consist of less than half of electrons in their outermost shell. On the behalf of this characteristic, metals do not form compounds with each other. Combine with nonmetals that have more than half the maximum number of valence electrons. The difference in metal generally due to their chemical reactivity[48], [49]. Most reactive metals are lithium, potassium, and radium, while those which have low reactivity are gold, silver, palladium, and platinum metals.

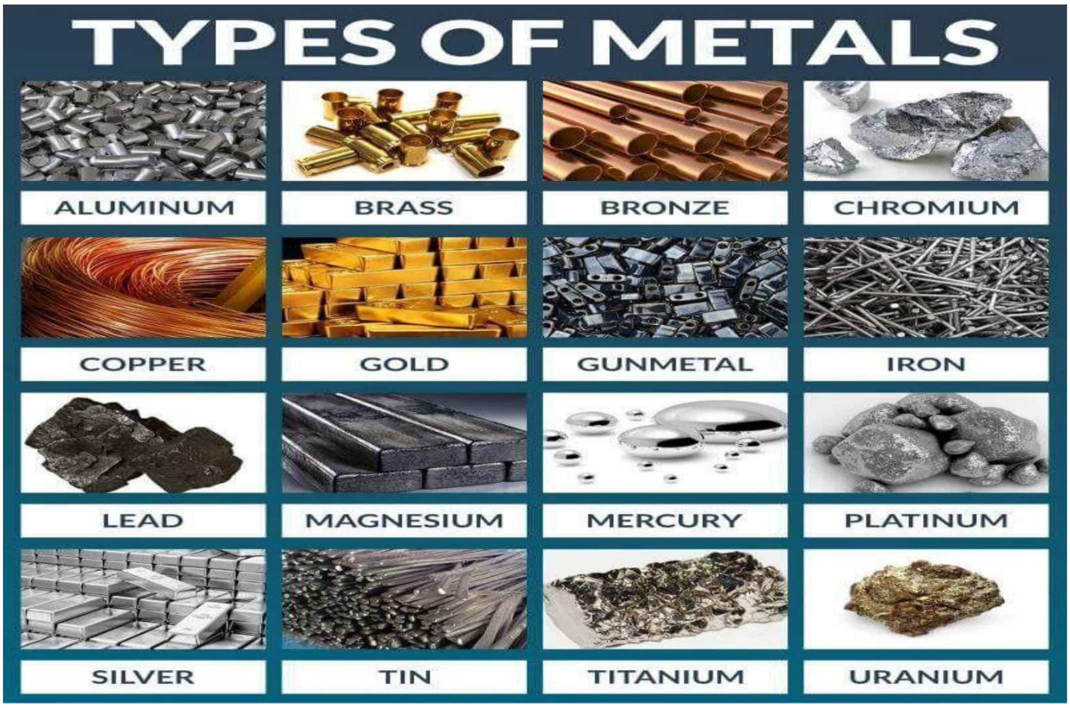


Figure 1.6 Different type of metals [48]

1.5.1 Metal Dust

When operations are performed without lubricant the grinding or sanding media breaks small pieces of metal-free from the metal surface to produce a smooth surface. Even very smooth surfaces are still likely to look like grooves and mountain ranges under magnification. Further sanding with fine media knocks the tops of those mountains, making the hills, with the resulting particles becoming finer and finer like dust. In this case, grinding steel results in the fresh iron surfaces to oxidize (burn) rapidly when in contact with the oxygen in the air. The metallic dust in this case will include metal oxides.

Maximum amounts of exposure of metal dust can irritate your skin, ears, eyes, throat, nose, and cause lung damage. Symptoms of congestion, cough, rash, or trouble breathing. Inhaling these metals can damage the nervous system, lungs, and other organs, like kidneys and liver. Chromium and cadmium metals have been a concern with cancer[50], [51]. Even at little low exposure levels cause sleeping problems and depression. Heavy metals exposure results in several health concerns, like vision problems, headaches, mental confusion, allergies, neurological impairment (lead), short-term memory loss, chronic fatigue, muscle and joint pain, gastrointestinal issues, and kidney failure. A person can be treated by a physician but the toxicity effects not are fully reversible.

Figure 1.7 Metal dust [50]

1.5.2 Metal Properties:

An element is defined as a substance composed of one type of atom. Like the helium is built by helium atoms. Elements classification as metals or nonmetals even some elements have both characteristics are called metalloids. Three properties of metals are:

1. Luster:
   - When metal cut into pieces its surface looked polished and shiny.
2. Malleability:
   - Metals are malleable and can easily bend or shape. Smits from centuries used to make the equipment by heating metals and changing shape by using hammers. Most metals are used to make wire due to their ductility.
3. Conductivity:
   - Metals show good conduction for heat and electricity Because of ductile nature which is used for ideal electrical wiring.

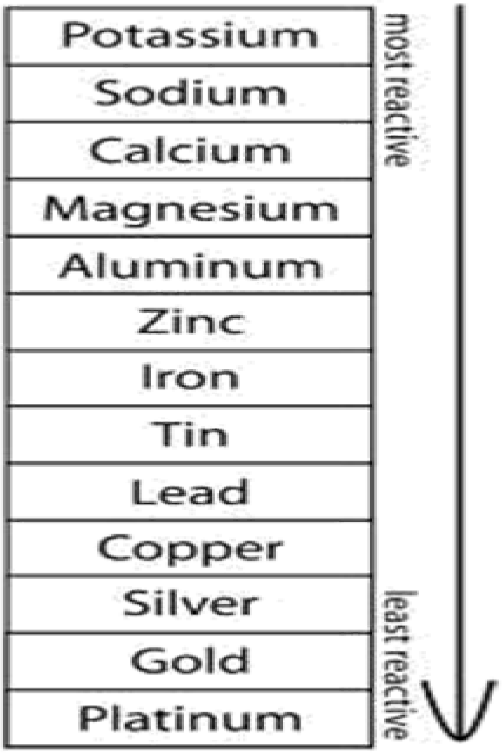


Figure 1.8 Most/Least reactive metals[52]

Table 1 Emissivity Table of Metal and Non-Metallic materials [53]

Metal	Emissivity	Non-metal	Emissivity
Bare aluminum	0.02–0.4	Concrete (rough)	0.93–0.96
Gold	0.02–0.37	Glass	0.76–0.94
Copper	0.02–0.74	Wood	0.8–0.95
Lead	0.06–0.63	Carbon	0.96
Brass	0.03–0.61	Human skin	0.98
Nickel	0.05–0.46	Paper	0.7–0.95
Steel	0.07–0.85	Plastic	0.8–0.95
Tin	0.04–0.08	Rubber	0.86–0.94
Silver	0.01–0.07	Water	0.67–0.96
Zinc	0.02–0.28	Sand	0.76–0.9

1.6 Polymer Films

Normally, the layer thickness of polymeric film is up to 0.2–0.3 mm thick. Polymeric thick layers are called sheets. The most common group is cellophane. A second, large group of polymeric films are made of artificial polymers, or products of the chemical treatment of natural polymers[54], [55]. Films produced in this group from natural rubber and cellulose esters which undergone prior hydrochlorination. A synthetic polymer is used for the Largest group of polymer film formation that is commonly based on polyvinyl chloride (PVC), polyolefins, polyvinylidene chloride, polyamides, polystyrene, polyimides, and polyethylene terephthalate[56], [57].

Methods for the production of polymer films are.

- Extrusion [58], [59].
- Casting [60].
- Calendaring[61].
- Polymer solution casting

Polymer solution casting

Polymer solution casting is the process which produces high quality film with superior electrical, mechanical and optical properties as compared to extrusion process. In this process, first the polymer is dispersed in the solution then solution is coated on the substrate followed by the drying of the solvent to produce a solid layer on the surface of the substrate. Afterwards, the solid layer is peeled off from the substrate to produce final film. Multilayer products can also be produced by this process. Several manufacturing advantages of polymer solution casting over extrusion methods include:

- Low temperature processing as compared to traditional methods.
- High-temperature resistant films
- Simplified incorporation of additives and fillers.
- Single pass manufacturing of multi-layer films

- A wider range of material choices

Advantages of the resulting film include:

- Uniform thickness.
- A wider range of film thicknesses, from 150 μm down to less than 12 μm.
- Dimensional stability.
- Films that are gel and pinhole-free.
- Excellent flatness.
- Isotropic orientation
- Absence of typical extrusion process lubricants.

1.7 Cellulose Acetate (CA)

CA was first prepared in 1865. CA is employed as a frame material for eyeglasses, film base in photography, as a component in some coatings, also used as a synthetic fiber within the manufacture of playing cards and cigarette filters. In the photographic film, CA replaced nitrate film in the 1950s, which is far less flammable and cheaper to produce[62]–[64]. Cellulose ester has come from the acetylation of cellulose. Cellulose in its original form is digested by a sizable number of microorganisms because it's a familiar natural product. The introduction of acetyl groups makes an important change in its biostability. CA is usually known as a biodegradable polymer[65].

Acetate is valuable manufactured fiber and cheap, has good draping Acetate is employed in fabrics like brocades, satins, and taffetas to intensify drape, luster, and body.

Hand: soft, smooth, crisp, dry, and resilient

Comfort: Quickly, wicks, breathes.

Color: Deep brilliant shades

Performance: Colorfast to dry cleaning, colorfast to perspiration staining.

Tenacity: Weak fiber with breaking tenacity

Luster: light reflection creates a signature appearance

Heat retention: Poor thermal retention

Abrasion: poor resistance

Dye ability: Cross-dying method where yarns are woven into fabric in the desired pattern; Solution-dying method provides excellent colorfastness.

Chapter 2

Literature Review

There is a high demand for glass buildings and windows for both residential and commercial use. This built high land costs in urban centers as well for aesthetic and comfort reasons. Materials selection for such buildings is by the mechanical limits. As extensive glass is a prominent feature in new architecture for more than aesthetic reasons. The functional materials and energy windows have become a subject of study towards energy efficiency that has been discussed aplenty. There is several coating technologies for example low emission (low e) coating or low e films demonstrated for energy saving windows. The passive infra-red regulating coating for smart windows application.

Among all the elements of building fabric, windows, and glazing units contribute heavily to the energy consumption of the building. Buildings energy loss is up to 60% due to windows. The performance of a window is assessed by several parameters including total solar energy transmittance (g-value), thermal transmittance (U-value), and air leakage. Compared to other building elements, windows have a remarkably higher U-value.

However, due to their role in day lighting and ventilation and their psychological impact on occupants, windows are inevitable. In recent decades, efforts have been made to improve the thermal performance of glazing systems through new technologies, among which, window films impose the least disturbance to occupants as a retrofitting measure.

Publication	Research Work Overview	Materials & Method	Properties	Application
Amun Amri et al. **[66]**	Huge demand for less cost and environment friendly techniques for making good quality characteristics. A comprehensive study of those characteristics is an important component within the optimal design of SSA coatings.	**Materials** • Solar selective absorber (SSA) coatings • Copper oxide-based absorber • Cobalt oxide-based absorber **Method:** Sol-gel	• Absorb incoming radiation through re-radiation • Minimal thermal energy loss • Excellent durability • High absorbance • Overcome by design parameters, heating temperature, or concentrations during the synthesis.	• This paper is to improve technical issues and developments review of sol-gel methods • Solar collectors
Lukas Kinner et al. **[54]**	Al-doped ZnO DMD electrode works beyond with the extraordinary for transparency on PET.The expansion of the TiOx interlayer on the flexible substrate, rendering almost like its growth on glass. This increases the transmittance of the DMD electrode and decreases sheet resistance	**Materials** TiOx/Ag/AZO electrode on PET substrate **Method:** Sputtering	• High flexibility • Dense film growth • High performance ITO free electrode • Intermediate transmittance more than 85% almost range 400–700 nm apart from this sheet resistance below 6 Ω/square	• Photovoltaic and optoelectronic applications. • The article provides a detailed clarification of the problems related to the reactive sputter-deposition of TiOx on PET

Ann-Louise et al. [67]	In advanced glazing, thin films show many prospects. That can achieve by additional functionalities such as power generation, self-cleaning	• Functional metal oxide thin films • CeO_2 and TiN with TiO_2 by **sol gel process** • Al or Ga doped ZnO films by **CVD method**	• Hydrophobic self-cleaning properties • Excellent aesthetic • Good thermochromic performance	• Self-Cleaning Glazing Applications. • Solar Cell Glazing Application • Dye-Sensitized Solar Cells (DSSC) • Organic Photovoltaics (OPVs)
Jin Young et al. **[44]**.	Low-emissivity coatings were coated onto flexible substrates at room temperature by using amorphous semiconductor materials	**Materials** Si–In–Zn–O (SIZO) and metal Ag, oxide–metal–oxide OMO **Method:** Sputtering	• Excellent electrical, optical and emissivity properties. • Excellent transmittance and electrical resistance • Good adhesion • Excellent flexibility	• Energy-Saving Window Applications • SIZO–Ag–SIZO is good for high performance and cost-effective low-E coating fabrication.
N. Abundiz-Cisneros, et al. **[68]**	Investigate alternative metallic coatings to replace Silver in Low-E glass filters Replacement of standard Low-E filters are based on a Silver with Al based filters	**Materials** • Al Low-E filters • $TiO_2/Al/Al_2O_3/TiO_2$ **Method**: Sputtering	• Cost Effective • Better performance in the infrared than the one-layer Ag-based filter • Transmittance is as good as a commercial three-layer Ag filter	• Architectural glass pane. • Simulation and experimental validation of multilayer filters

Cheng Zhang et al. **[70]**	Efficient flexible-printable optoelectronic devices are fabricated in the absence of ideal flexible transparent conductors with Ulta super optical, electrical, and mechanical properties	**Materials** Ag-based thin films doped with Al **Method** Co-Sputtering	• Ultra-smooth surface • Visible Transmittance of 80% without any antireflection coating • Sheet resistance less than 20 Ω sq−1, • Excellent mechanical stability over 1000 bending cycles. • Ultra-thin • Stability at elevated temperature	• Window Applications • Optoelectronic • Nanophotonic devices

2.1 Aims and Objective:

- ✓ Synthesis and characterization of flexible polymeric low emissivity films for glass/windows application to reduce the energy loss from buildings.

2.2 Research Project work:

The research in this thesis is based on low emissivity polymeric films incorporated by metal dust by a simple solution casting method. The polymer as a substrate is cellulose acetate (CA) for film casting. Due to their good durability; acetate films are also widely used. CA is biodegradable and generally recognized as safe. Fabrication of these cellulose acetate base polymeric films begin for low cost, easy access, and environment friendly for the use of metal in a form of dust incorporated in the polymeric film that relates to solar window films for controlling the influx of solar radiation through windows, and more particularly relates to an improved window film having low emissivity, excellent durability, and energy efficiency. The thin films are supported over a glass or polymeric substrates to gain efficient properties and phenomena by them, so the relatively low emissivity coefficient makes aluminum and tin suitable product for limiting the radiated heat from a body.

Chapter 3

Experimentation Methods

3.1 Materials Selection

- Metal Dust (Al, Sn)
- Acetone (As a Solvent)
- Cellulose Acetate (Film Substrate)

3.2 Deposition of Thin Films

The thin films are supported over a glass or polymeric substrates to gain efficient properties and phenomena by them. Substrates affect the properties of thin films. The deposition of thin films can be done by using various techniques in which vacuum is used.

Continuous solvent cast is one of the oldest methods for plastic film manufacturing developed more than hundred years ago but after 1950 extrusion techniques were used for the productions of plastic films and solvent cast technology has declined. Nowadays, solvent cast technology is becoming increasingly attractive to produce films with extremely high-quality requirements. The advantages of this technology include uniform thickness distribution, maximum optical purity, and extremely low haze. The optical orientation is virtually isotropic and the films have excellent flatness and dimensional stability. The cast film can be processed in-line with an optical coating design. The tremendous growth of new liquid crystal display applications has incited the development of new materials and improved processes for solvent casting and coating techniques.

Table 2 Material Concentration for Low E Film Fabrication

	Solution A		Solution B	
Low Emissivity Films	Acetone	Cellulose	Acetone	Metal-Dust
Al/CA,Sn/CA-Films (10%-loading)	15ml	0.9g	5ml	0.1g
Al/CA,Sn/CA-Films (5%-loading)	12ml	0.95g	3ml	0.05g

3.3 Synthesis (Experimental Procedure)

The formation of low emissivity film depends upon three main chemicals in this research work. A polymer as a substrate that is cellulose Acetate average (Mn ~50,000, Molecular formula: C164H174O111, Molecular weight: 3921.1 g/mol) Commercial Metal dust Aluminum (Al), Tin (Sn) based on its emissivity values and Acetone as solvent (Empirical formula: C3H6O, Molecular Weight: 58.08) used for film casting by purchasing from vendors Sigma Aldrich.

Method details

The Al/CA (Aluminum base polymeric film) and Sn/CA (Tin base polymeric film) were synthesis individually with 5 %, 10 % at different concentrations was prepared by the solution casting method. The Cellulose acetate CA was procured from the market in the granular form. The solution prepared for film casting into different steps. AL/CA film with 10% loading the steps are given below first solution A in which CA 0.9 g is added slowly into 15ml Acetone and separately a solution B in which 0.1 g of Al Metal dust into 5ml Acetone now put both solutions A and B on stirring for 24 hrs. Now pour solution B into solution A rapidly. That is a combined solution of A and B called a mixed solution that term as solution C.

Then the next step is sonication for this use bath sonicator and sonicates a solution C for 120 mins and 30 mins degas in an airtight media bottle to avoid bubbles in the film. The next step is on casting after sonication left the solution C for 30 mins then the resultant solution was then poured on to a plain cleaned Petri dish for film casting gradually and uniformly in an isolated chamber and cover it with a lid so there is no air contact as a solvent start to evaporate. Note that during casting solution spread homogeneously so that we get uniform thickness in film and the solvent was subsequently allowed to evaporate gradually throughout 12-24 hrs. in a dry atmosphere. The film was then physically peeled off from the surface mostly film peeled itself as a solvent evaporating itself. The cast surface area, the material quantity, as well as the density of the material can determine the film thickness. For Al/CA film 5% loading have the same steps but the concentration of solution A of 0.95 g CA into 12 ml Acetone. And for solution B 0.05g Al metal dust into 3ml Acetone. The Sn/CA thin polymeric film cast with 5-10% loading has the same steps and concentrations. The fabrication and Casting process of Low emissivity films is shown.

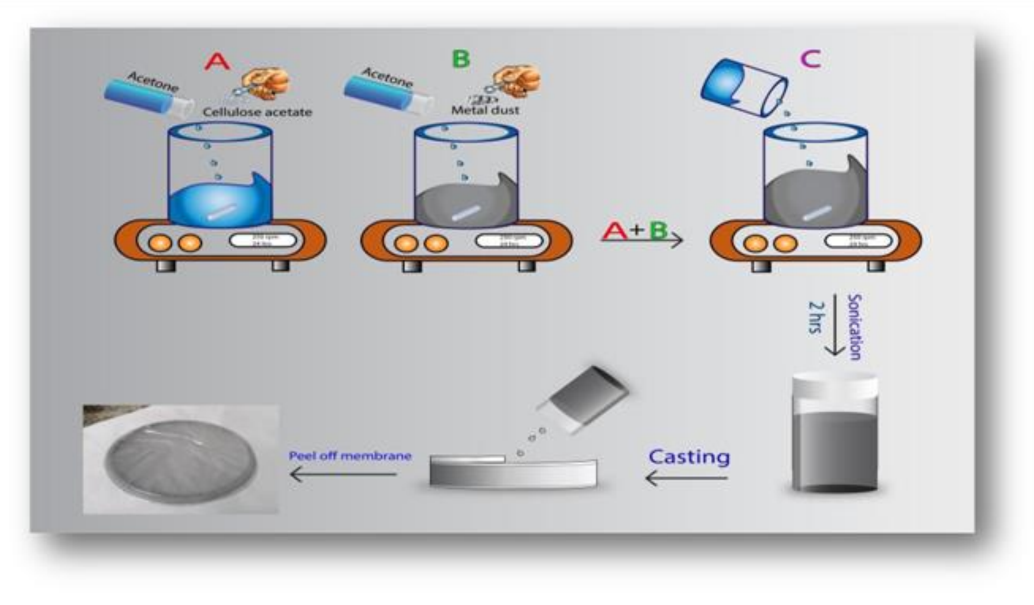


Figure 3.1 Fabrication of low Emissivity film incorporated with Metal dust

CHAPTER 4

Introduction to Characterization Techniques

4.1 Scanning Electron Microscope (SEM):

In this technique, the fine beam of electrons is focused over a specimen's surface. Photons or electrons are knocked off from the material's surface in the result. These knocked off electrons are then focused on the detector. The output from the detector modulates the brightness of the cathode ray tube (CRT). For every point where the electron beams are focused and interact, it is plotted on the consequent point on CRT and the material's image is produced. The electron-surface interaction causes the release of secondary electrons (SE), backscattered electrons (BSE), and X-rays [71].

The common SEM mode for detection is via secondary electrons. These electrons are emitted from near the sample surface. So, a pronounced and clear image of the sample is obtained. It can reveal the sample in detail. Also, the elastic scattering of incident electrons takes place and release backscattered electrons. They emerge from deeper locations as compared to secondary electrons. So, their resolution is comparatively low. When an inner shell electron knocks off from its shell it emits characteristic x-rays We use SEM as it has easy sample preparation and we can figure our sample‘s morphology, chemistry [72]. Magnification of SEM can be controlled from 10 to 500,000 times. Morphology of the materials was examined (JEOL-JSM- 6490LA).

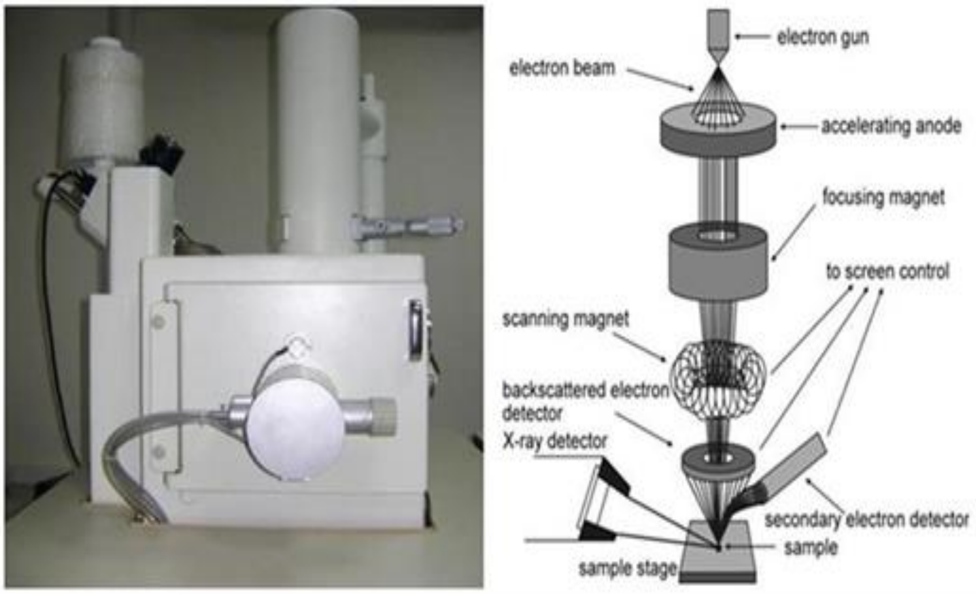


Figure 4.1(a) JOEL JSM-6490LA present at SCME; (b) SEM Schematic

4.2 X-ray diffraction (XRD)

It is used for the crystal structure determination of the material. It is a non-destructive technique, and it provides fingerprints of Bragg‘s reflections of crystalline materials[73]. It consists of 3 main parts. A cathode tube, sample holder, and detector. X-rays are produced by heating filament elements that accelerate electrons towards a target which collide with the target material with electrons. Crystal is composed of layers and planes. So, x-ray which has a wavelength having similar to these planes is reflected that that angle of incidence is equal to the angle of reflection. ―Diffraction‖ takes place and it can be described as by

Bragg’s Law: $2d\sin\theta = n\lambda$

λ is the wavelength of the x-ray,

d is the spacing of the crystal layers (path difference),

θ is the incident angle (the angle between the incident ray and the scattering plane)

n is an integer

Constructive interference takes place when Bragg law is satisfied and Bragg reflections‖ will be picked up by the detector. These reflections' positions tell us about inter-layer spacing-ray diffraction tells us about the phase, crystallinity, and sample purity. By this technique, one can also determine lattice mismatch, dislocations, and unit cell dimensions. X-ray diffractions were performed by the STOE diffract meter at SCME-NUST.

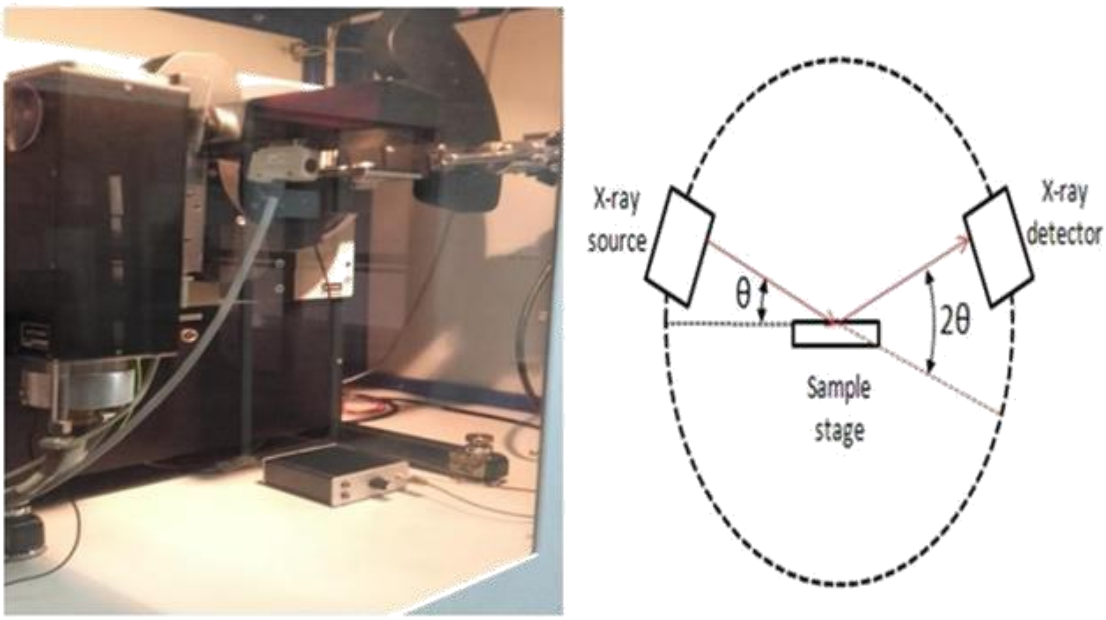


Figure 4.2 XRD present at SCME- NUST (b) XRD basic

4.3 UV-VIS-NIR Spectroscopy

UV-Vis-NIR spectrophotometer UV-3600i Plus incorporates the latest technology in optical components to mark high resolution, sensitivity, and an ultra-low light level, for new solutions. Measurement capacity of the UV, Visible, and up to 3300nm in the near-IR allows all types of samples including solids, powders, wafers, films, and liquids. Applications include but are not limited to haze analysis, band gap, photovoltaic, coatings, and optical component characterization.[74]

The equipped UV-3600i Plus with double mono-chromatics grating to achieve ultra-low light levels that confirms constant optical resolution of 0.1 nm or less in the ultraviolet and visible region and 0.4nm for the near-infrared region. The wavelength range span from 185 nm to 3,300 nm making the spectrophotometer suitable for all kinds of samples, especially samples encountered in the field of materials that absorb or reflect in the ultraviolet, visible, and near-infrared regions[75], [76].

UV-Vis-NIR Shimadzu Lab Solutions control software is added with the instrument. Measurement modes are photometric, spectrum, quantitation, kinetics, time course, and bio-methods.

Figure 4.3 UV-Vis-NIR spectrophotometer in USPCASE, NUST

4.4 Thermal Radiometer unit

The laws of heat transfer by radiation are measured by this apparatus using two alternative energy sources one is the light source and the second is the radiant heat source [77]. The radiation sources mounted on a horizontal aluminum profile base frame with a measuring scale. Heat source temperature can be controlled and measured by a radiometer. Metal plates with the different polish surfaces are fitted with thermocouples to demonstrate the effect of emissivity on radiation emitted and received.

Base frame Material: Twining aluminum profile

Temp range: 200-6000 K

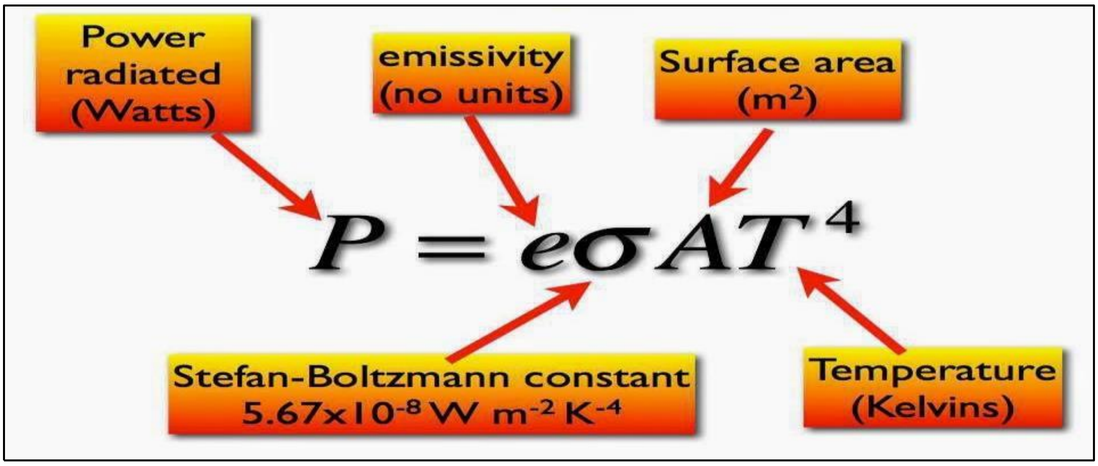
Two black radiation plates, one gray and one polished surface, all with base clamp Insulated adjustable aperture plates 2.

Light source Light bulb Filters with different opacity and base clamp

Data display software and analysis by computer (separately supplied) whereas the Power supplies 220 V, 1 Ph, and 50 Hz.

U-VALUE

Heat transmittance through the coating is measured by the thermal transmittance or U-value (U). U value measurement of heat flux through glass slides divided by temperature change into both sides of samples and its unit is W/m2K. The lower the value the better is the insulating properties of the coating. By using Stephan's Boltzmann law emissivity is measured



The mathematical formula of U value is given as:

U-VALUE = Radiometer flux / Temperature Difference

However, windows consume high solar energy due to their higher U-values. However, there is a need to reduce the total energy gained by the windows. The U-value is calculated by measurement of heat radiation transfer through the coated samples. A heat source is employed at a temperature of 673 K on one side of the sample. The heat flux transferred through the sample is measured on the other side by the radiometer. The heat flux density is then divided by the difference in temperatures measured on both sides of the sample.

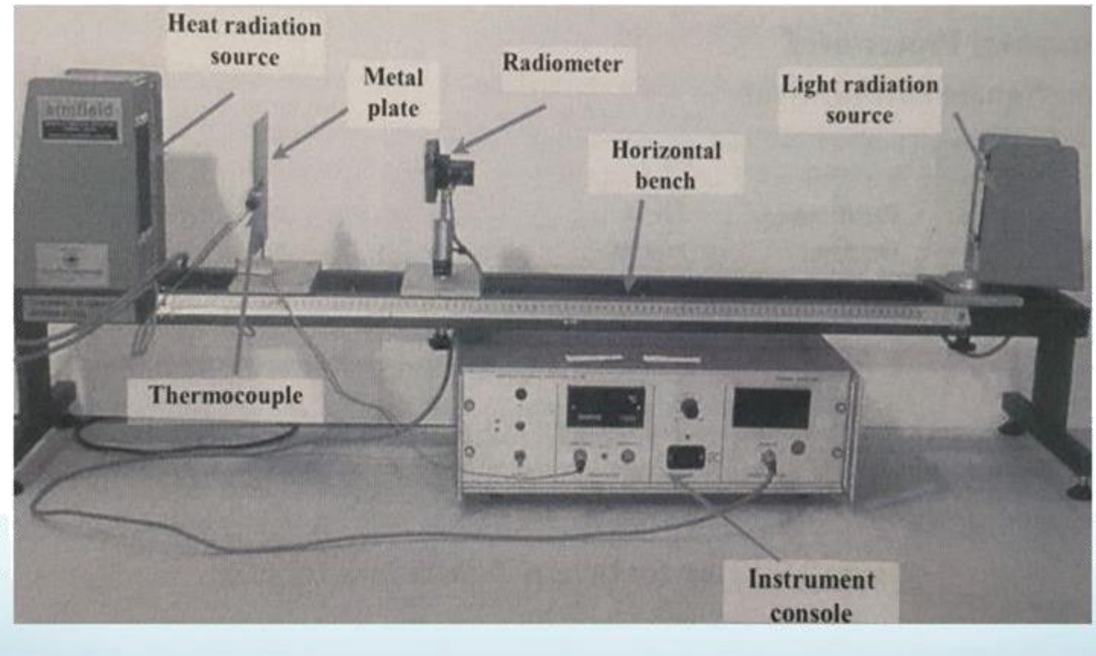


Figure 4.4 Thermal Radiation Unit-Control Unit Lab SCME-NUST

4.5 Contact Angle Measurement

It is a technique used to determine the contact angle of a droplet on a surface. A water drop is dropped on a sample surface and then is measured through different means. Here we use Drop Shape Analyzer DSA-25 shown in **Figure 4.5** present in SCME, NUST.

Drop Shape Analysis is an image study technique for defining the contact angle from the shadow image of a sessile drop. A drop is dropped onto a solid sample (sessile drop). An image of the drop is recorded with the help of a camera and transferred to 46 the drop shape analysis software. Outline recognition is primarily carried out based on a grey-scale analysis of the image. In the next step, a geometrical model telling the drop shape is fitted to the contour.

Figure 4.5 Drop Size Analyzer DSArop Size Analyzer DSA

4.6 Tensile Testing Test:

Linkam TST350 accurately Characterize the tensile properties of your sample relative to temperature utilizing the 0.001 N sensitive force transducer and capture high resolution images of the structural changes. Linkam TST350 Stage allows you to capture high resolution images of the structural changes. The TST350 is built with two precision ground stainless steel lead screws to maintain perfect uniform vertical and horizontal alignment. Sample jaws move in opposite directions to maintain sample in both reflected and transmitted microscope fields of view.

The sample chamber is gas sealed and can be controlled with various gases via the gas valves built onto sides of the stage, as well as combined with our RH95 Humidity Generator to test samples under different humidity levels.

Figure 4.6 Tensile stress testing Machine Linkam TST350

CHAPTER 5

Results and Discussion

The newer version of Low-E window film tends to have a more transparent film compared to standard solar control window film, allowing natural light while minimizing heat. Film thickness depends on the viscosity of the solution. The petri dish must be on an even surface and this will control the uniformity of the film and a also the way you pour the solution on the petri dish, try to make possible that casting can be done under a highly isolated environment to avoid impurities after trying a couple of times get uniform films. The low emissivity films fabricated in this research meet the best efficiency criteria discuss in this chapter as final low emissivity film mentioned below in figure 5 . 1 which is low emissivity polymeric composite films incorporated by metal dust.

Figure 5.1Low emissivity polymeric film incorporated by metal dust.

5.1 Scanning electron microscopy (SEM Analysis)

The morphology of all the prepared samples was examined by SEM analysis**. Figure 5.2** shows the SEM of the Al and Sn metal dust. The average particle size as measured from SEM is found to be 1.667 μm and 2.13 μm for Sn and Al respectively.

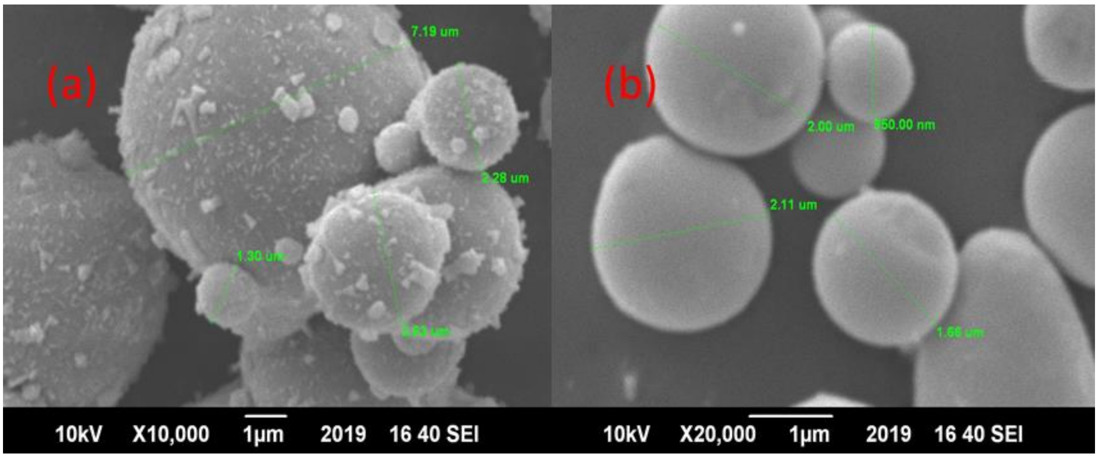


Figure 5.2 SEM images of dust used in films fabrication (a) Al (b) Sn

Tinted film with little haze low emissivity films were obtained by solution casting. All films showed the color of in cooperated metal dust into the polymer. **Figures 5.3 (a-f)** shows SEM images of CA/Al-10%. **Figure 5.3(a)** shows Al/CA with 10% loading is a very dense film with bubbles because of air contact whereas other film is uniform dust Particle distribution and no bubbles and no cracks because this film is casted in high isolated environment as mentioned in **Figure 5.3 (b)** shows CA/Al-10% at high magnification the size of the bubbles ranges from 7 μm to 10 μm. CA/Al-10% dense film, homogeneous nature with no crack, and some bubbles in **Figure 5.3 (c-d)** at low and high magnification**.** This is due to the solvent acetone which has low boiling liquid so easily evaporate at room temperature the cross-section of the same film at different magnification in **Figure 5.3 (e-f)** with a thickness of 22 μm. The films were cracked while dipped in liquid nitrogen to measure the thickness under SEM.

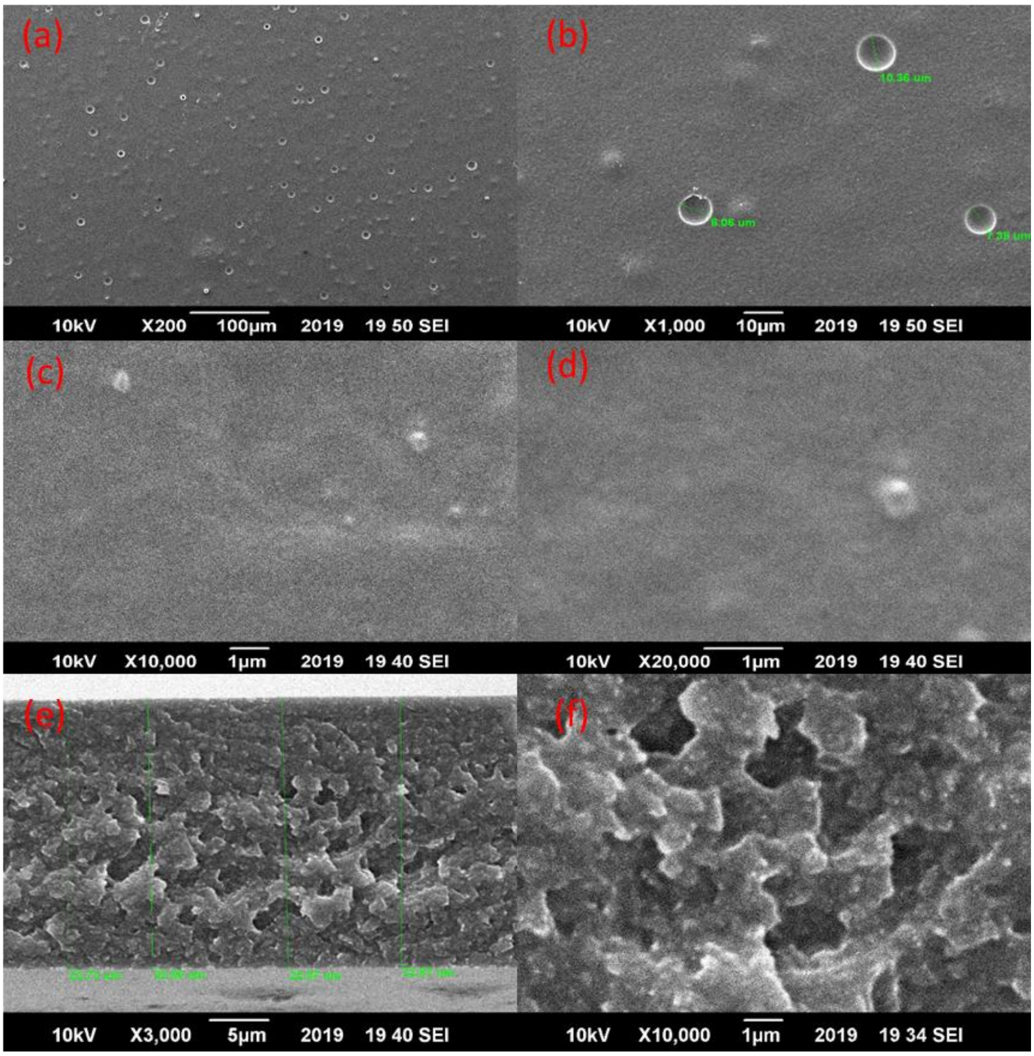


Figure 5.3(a-d) SEM images of CA/Al-10% at low and high magnification (e-f) Crossection images of CA/Al-10% at low and high magnification

Figure 5.4 (a-f) shows the SEM images and cross section of CA/Al 5% loading at low and high magnification. During casting under precaution, we get highly uniform, dense, tinted films with little haze due to dust incorporation in the film as shown in **Figure 5.4 (a-d)** whereas the cross-section images by dipping in liquid nitrogen and study under SEM having a homogeneous thickness in **Figure 5.4 (e-f),** all the film thickness depends on casting time and uniform coverage on petri dish the thickness of CA/Al-5% is 33 μm.

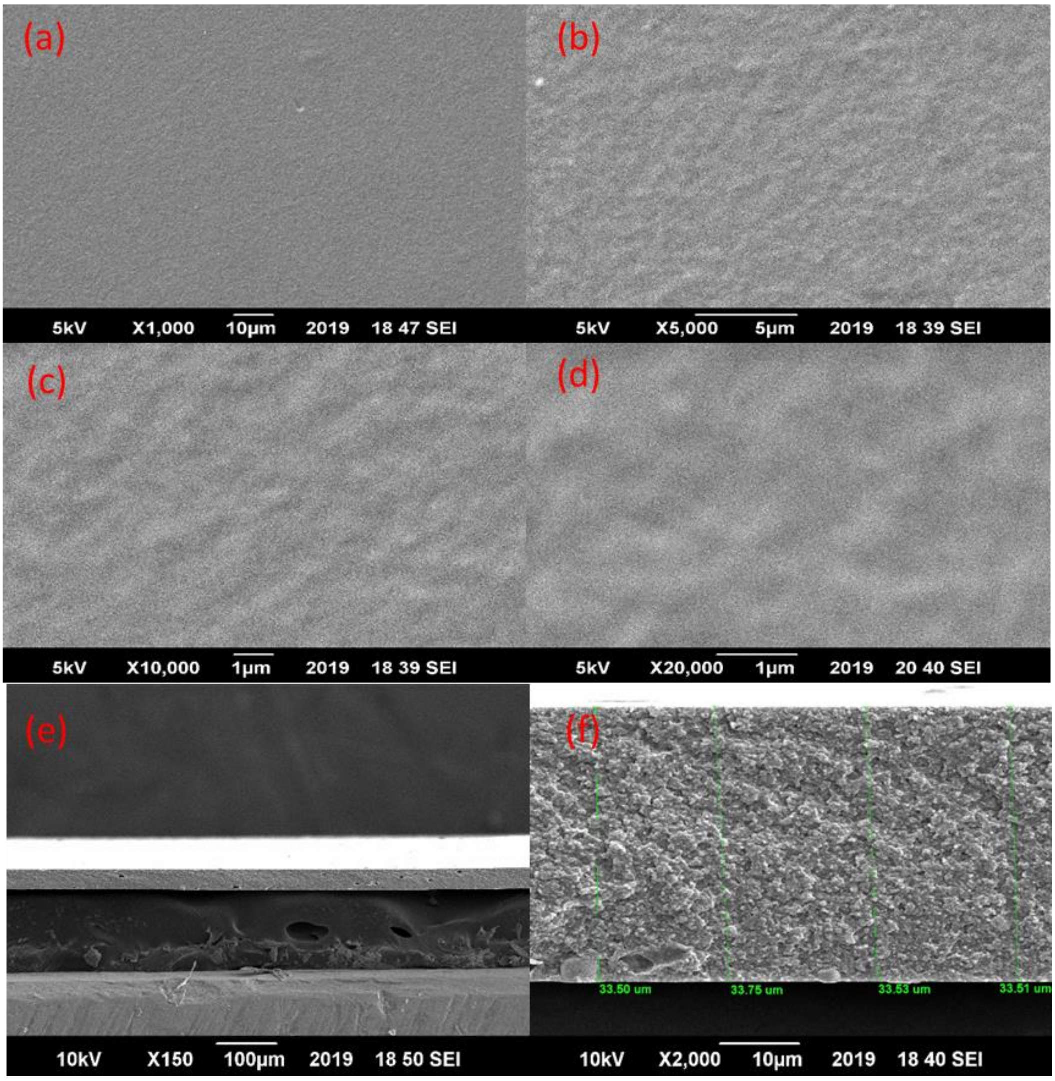


Figure 5.4(a-d) SEM of CA/Al-5% loading at low and high magnification (e-f) Crossection images of CA/Al-5% loading

Figure 5.5 shows SEM images of CA/Sn polymeric films at low and high magnification. **Figure 5.5(a)** shows a highly dense film of CA/Sn-10% loading with little bubbles with different range shows that film had an air contact and bubble due to acetone. Whereas more homogeneous and uniform film results in **Figure 5.5(b)**. Some dust on the surface of Sn/CA-5% loading with more homogeneous and dense appearance under SEM in **Figure 5.5(c-d).** The cross-section of CA/Sn-5% loading is also done by dipping the film in liquid nitrogen for some instant and then tear apart and place under SEM where the thickness is 33 μm is shown in **Figure 5.5(e-f).**

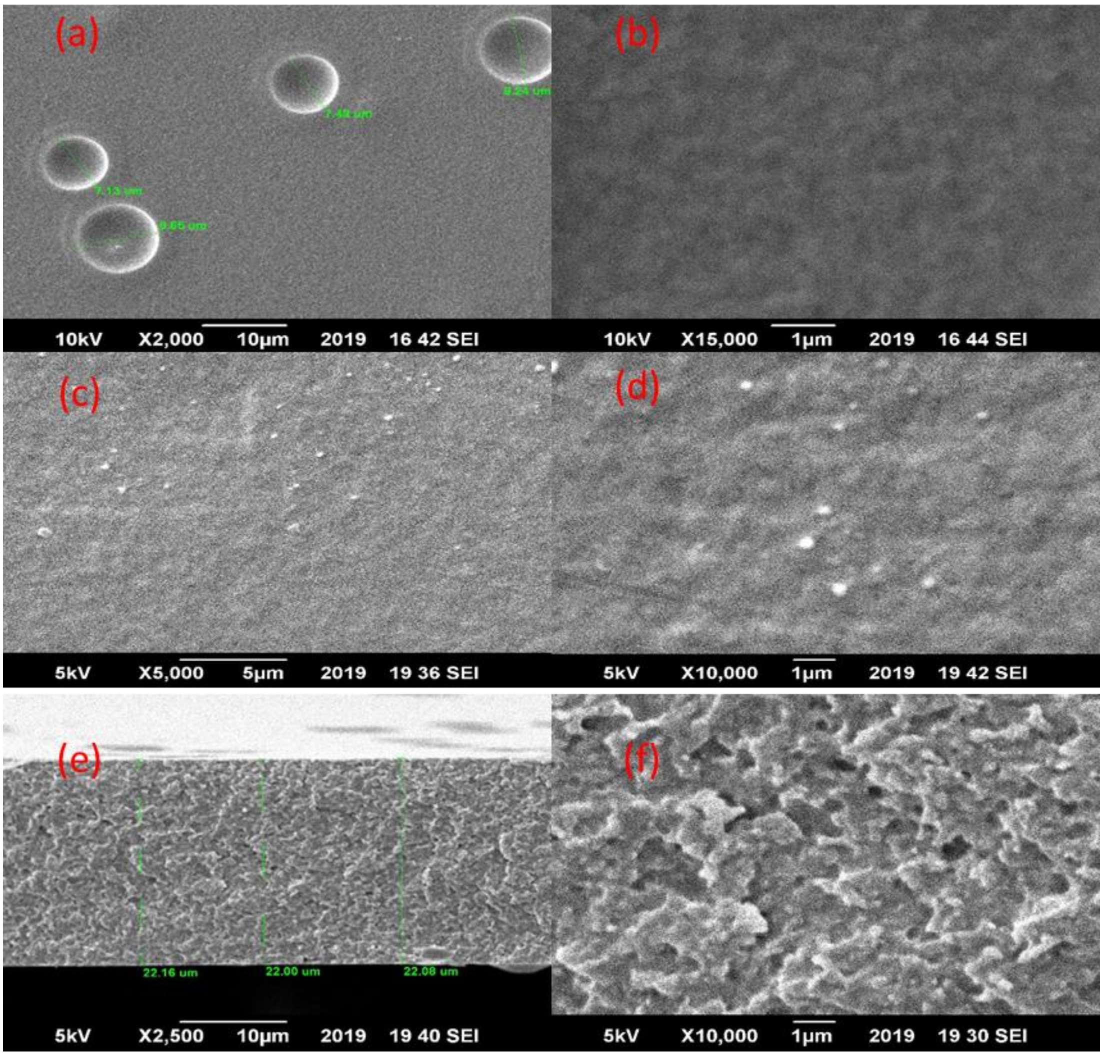


Figure 5.5 SEM images of CA/Sn polymeric films at low and high magnification (a-b) CA/Sn-10% (c-d) CA/Sn-5% (e-f) Cross-section images of CA/Sn 5%

5.2 X-Rays Diffraction Analysis (XRD)

The XRD has been performed to evaluate the phase and crystal structure of all the prepared films as shown in **Figure 5.6**. Pure CA analysis shows that a broad diffraction peak at 22.9º corresponds to JCPDS (00-003-0226). CA-Al 5% and CA-Al 10% samples show broad peaks of pure CA and sharp peaks of metal dust Al which confirms the addition of Al dust in CA phase corresponds to JCPDS (00-003-0226) having (101), (002), (200) planes and JCPDS 00-004-0787) having 111), (220) and (200) planes respectively. CA-Sn 5% and CA-Sn 10% samples show broad peaks of pure CA and sharp peaks of metal dust Sn which confirms the addition of Sn dust in CA phase corresponds to JCPDS (00-003-0226) having (101), (002), (200) planes, and JCPDS# (01-089-2958). Having (101), (211), and (321) planes respectively. The broadness of the CA peaks suggests its weak crystallinity and with the addition of metal dust crystallinity improved as well as the intensity and sharpness of peaks improved when loading of metal dust increased from 5 to10% for both Al films and Sn films.

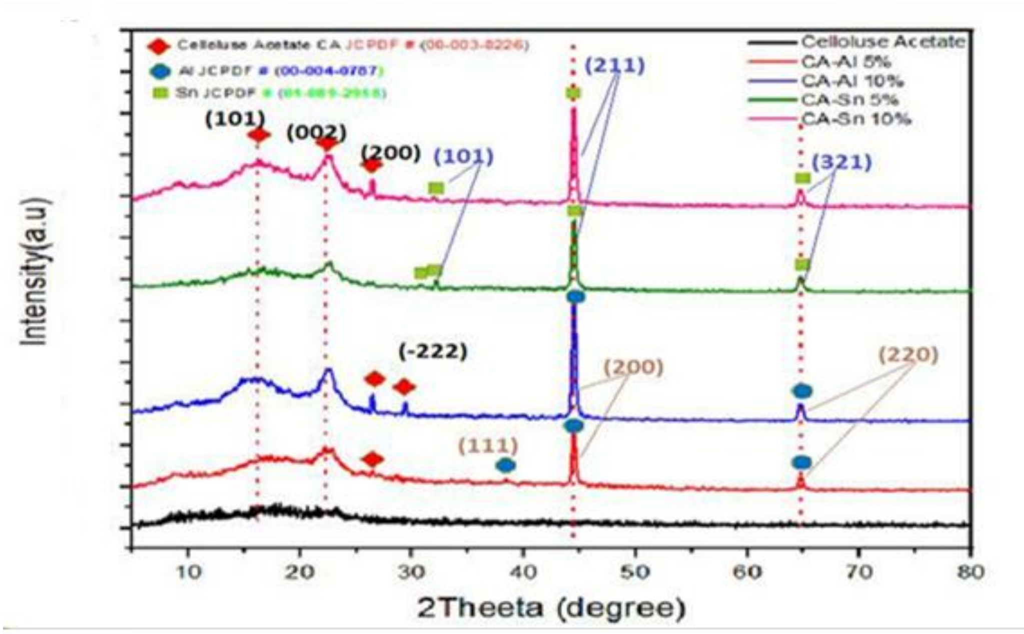


Figure 5.6 XRD analysis graph of Al/CA, Sn/CA, and pure CA polymeric films of different concentrations.

5.3 UV-Vis-NIR Spectroscopy Analysis

UV-Vis-NIR spectrophotometer is the range of wavelength that is used. Shimadzu UV-3600 Plus was used to measure the absorption, transmission, and reflection over a range of 200 nm to 3300 nm These fabricated low emissivity films have an energy efficiency of an air-conditioning system in a hot climate and of an indoor heating system in cold climatic. The reflectance characteristics of the pure CA, Al/CA, and Sn/CA films are visualized in **Figure 5.7** with different ratios of loading of metal dust. It is found that all low emissivity films based on Sn and Al was almost 80% reflectance with the reference of simple CA film.

As light was passed through all films of different wavelengths from 200 to 2500 nm in UV-Vis-NIR. The low-emissivity film shows partially transparent in (380–780 nm) visible region same as in the IR region this range (750-2500) is highly reflective to prevent heat losses. Same as relatively low reflectance in the Vis region and UV Region, meanwhile higher in the IR region, Reflectance rate in IR increases at 80% and then gently reduced to 70% and then 50% close to UV region corresponding to loading ratios of both Al and Sn films. Further, reduce 15% to 10% in the UV region. High level reflectance in IR and decreased sharply in the visible region toward UV- region which indicates the high reflectivity of radiant energy. **Figure 5.8** shows the absorption curve which indicated that some solar radiation can be absorbed by films in a range of 1% - 5% which means most of the radiation is reflected by Al and Sn based polymeric films as per requirement.

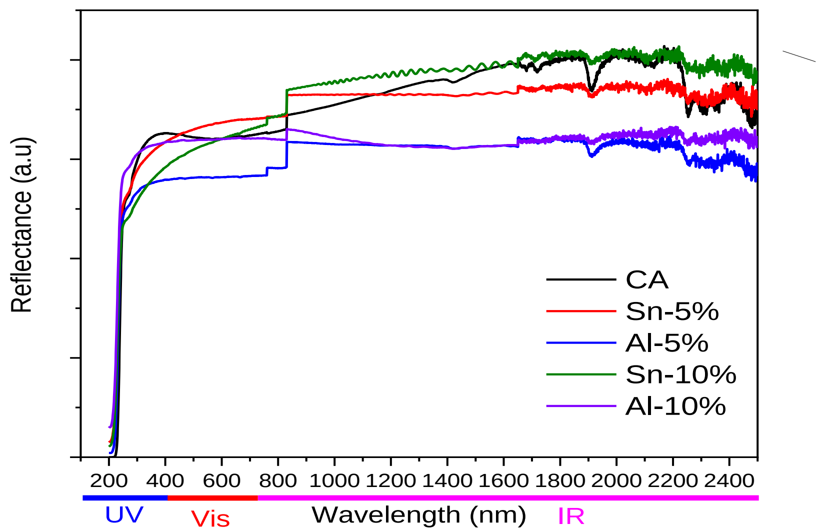


Figure 5.7 Reflectance curves of polymeric films of UV-Vis-NIR spectroscopy of Al/CA, Sn/CA, and pure CA films of different loadings

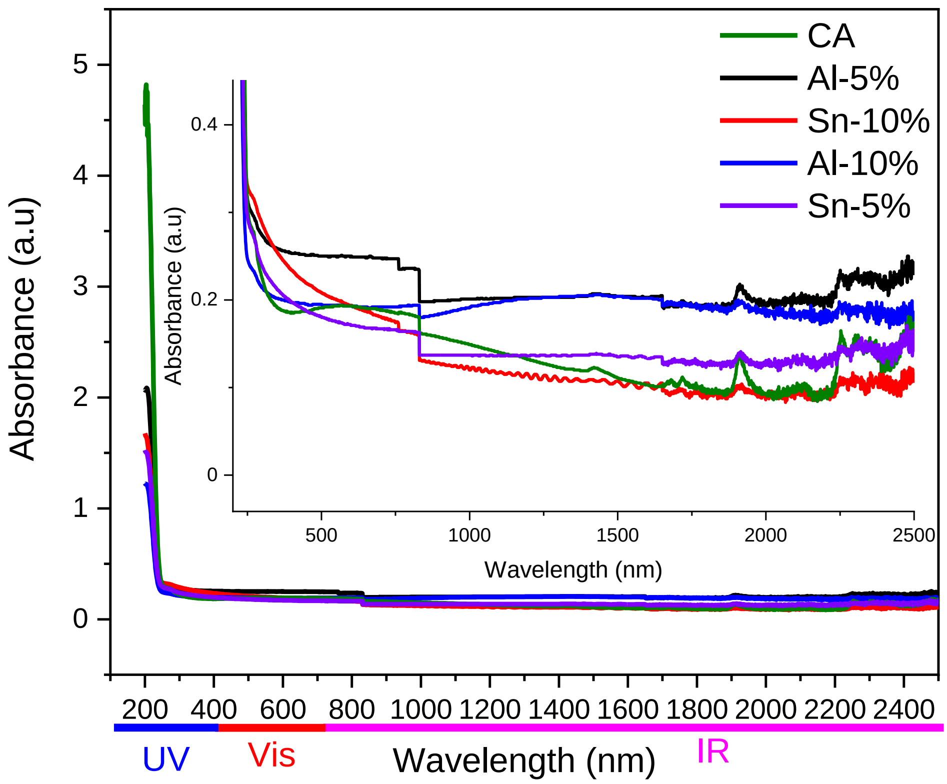


Figure 5.8 Absorption curves of polymeric films of UV-Vis-NIR spectroscopy of Al/CA, Sn/CA, and Pure CA films of different loadings.

5.4 Heat Radiometer Test:

The heat radiometer test relates to measure U-value which means emissivity value whereas U-Value show that how good a product prevents heat from escape. The lower U-Value, the greater a window's resistance to heat flow and the better its insulating value. The heat radiometer test is a heat test through which we measure U-Value of low e films by passing heat radiation of constant temperatures through the film in which heat comes from a source where the film is placed between the heat source and radiometer. The signals from the radiometer are received by the socket and showed value on the digital screen.

Through the heat test, we determined the U-value of simple glass, alone stand film, and the film with glass and adhesive. The formula and intake parameters for this test are:

U-Value (W/m^{2} . K) = *Heat flux Radiometer / Temperature difference*

- ✓ Distance b\w Sample & Radiometer = 25 cm
- ✓ Distance b\w heat source & Radiometer with sample = 35 cm
- ✓ Distance b\w sample & source = 25 cm
- ✓ Temp difference measured by Heat Gun at both sides of the film (T1-T2) in degree Celsius
- ✓ Standard temp for all measured reading by radiometer on Films = 300 ^{0}C

Table 3 Experimental U-Value of Low E Polymeric Films

Heat Flux on radiometer for all films	**23.2 W/m^2 K**	
Plain glass U- value	**7.26 W/m^2 K**	
Sample Film	**U-Value Alone Low-e Film W/m^2 K**	**U-value of low e Film Stand with Glass & Adhesive W/m^2 K**
Al/CA-10% loading	2.7+3.8 / 2 **= 3.2 ± 0.5**	2.7+3.3 / 2 **= 3.0 ± 0.3**

Al/CA-5% loading	3.0+3.9 / 2 **= 3.4 ± 0.4**	3.63+3.86 / 2 **= 3.7 ± 0.1**
Sn/CA-10% loading	2.9+3.3 / 2 **= 3.1 ± 0.2**	3.31+3.86 / 2 **= 3.5 ± 0.2**
Sn/CA-5% loading	3.5 + 4.2 / 2 **= 3.8 ± 0.3**	3.86+3.81 / 2 **= 3.8 ± 0.02**
Simple CA Film	3.8+ 4.6 / 2 **= 4.6 ± 0.4**	

5.5 Contact angle measurement:

Contact angle measurement was done to evaluate the nature of the all the prepared polymeric films as shown in **Figure 5.9 and 5.10.** DI water was used as a solvent to measure the contact angles over polymeric films. All the prepared Polymeric films show hydrophilic properties. Contact angle of pure CA film is 57^0.Moreover with the inclusion of metal dust Al and Sn to the CA film the angle is increased up to 10^0 due to increase in surface roughness as shown in **Figure 5.9 and 5.10**.

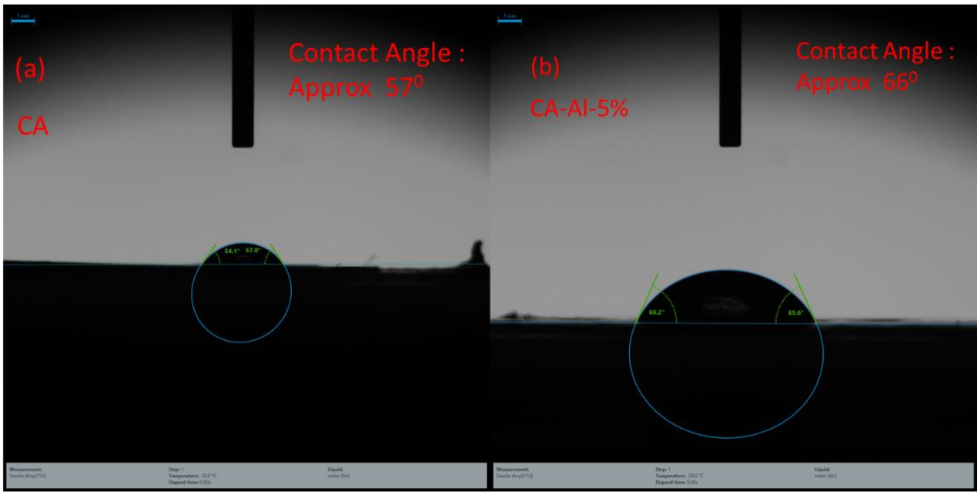


Figure 5.9 Contact Angle of polymeric films (a) CA (b) CA-Al 5%

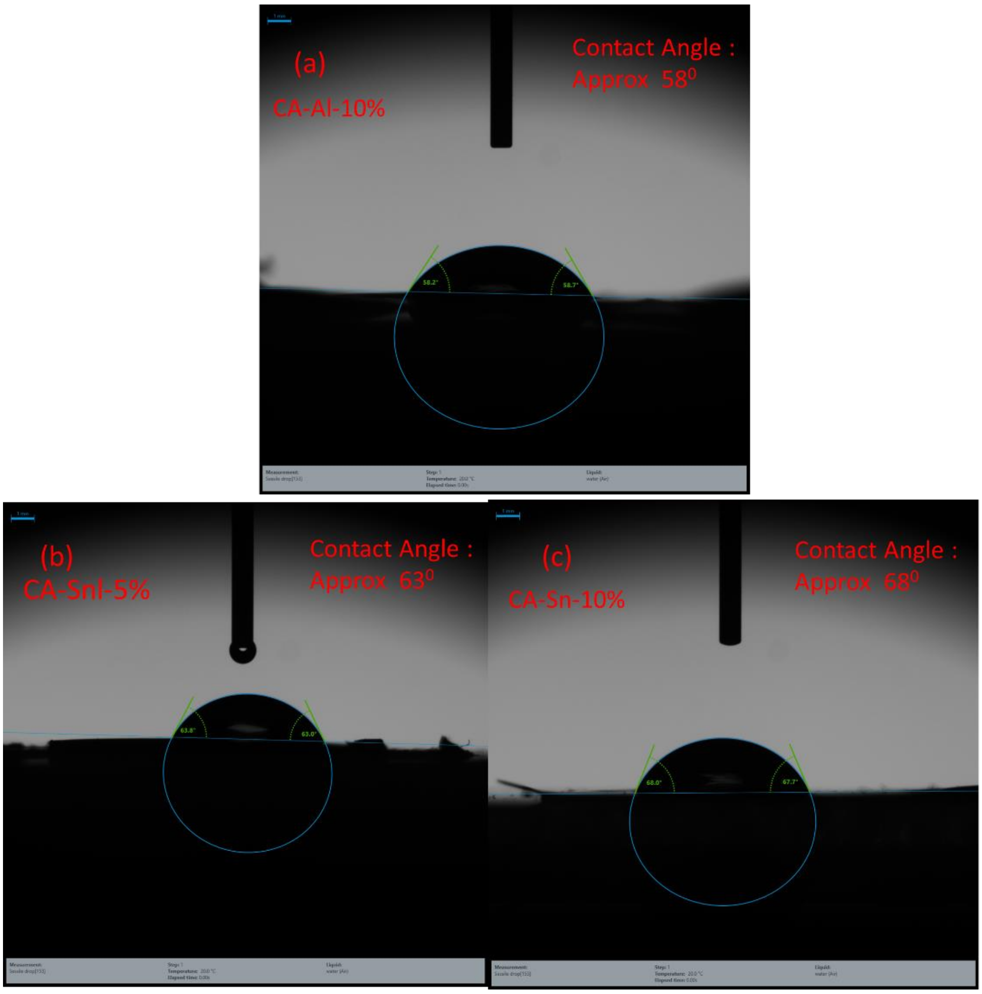


Figure 5.10 Contact Angle of polymeric films (a) CA-Al 10% (b) CA-Sn 5% (c) CA-Sn 10%

5.6 Mechanical Strength Testing:

Structural properties were analyzed by using tensile testing **linkam TST-350** on all the prepared samples of low emissivity polymeric composite films. As it is evident from stress-strain curves of all films in **Figure 5.11 (a,b,c,d,e)** with force 200N and strain rate 16.67 µm/s shows different strength performances due to different loading ratios of Al and Sn whereas the highest strength performance in the base sample CA. All the samples show different curves as shown in **Figures 5.11**.

In **Figure f** the bar graph shows the average tensile strength rate of all films in which CA shows higher strength performance that is about 18.75 MPa as compared to other films which shows 12.13 Mpa, 4.5 MPa , 9.26 Mpa and 10.9 MPa for Al-CA 5%, Al-CA 10% , CA-Sn 5% and CA-Sn 10% respect tively. It is evident from the graph that by adding metal dust into the base films it reduces the strength rate of all composite. The reason behind this result may be that Immiscible, immiscibility will result in the generation of larger spaces of CA to filled by metal dust at the interface which decrease the strength performance but we concluded that overall Low emissivity films have good strength performance which can greatly enhance the overall mechanical properties of these films.

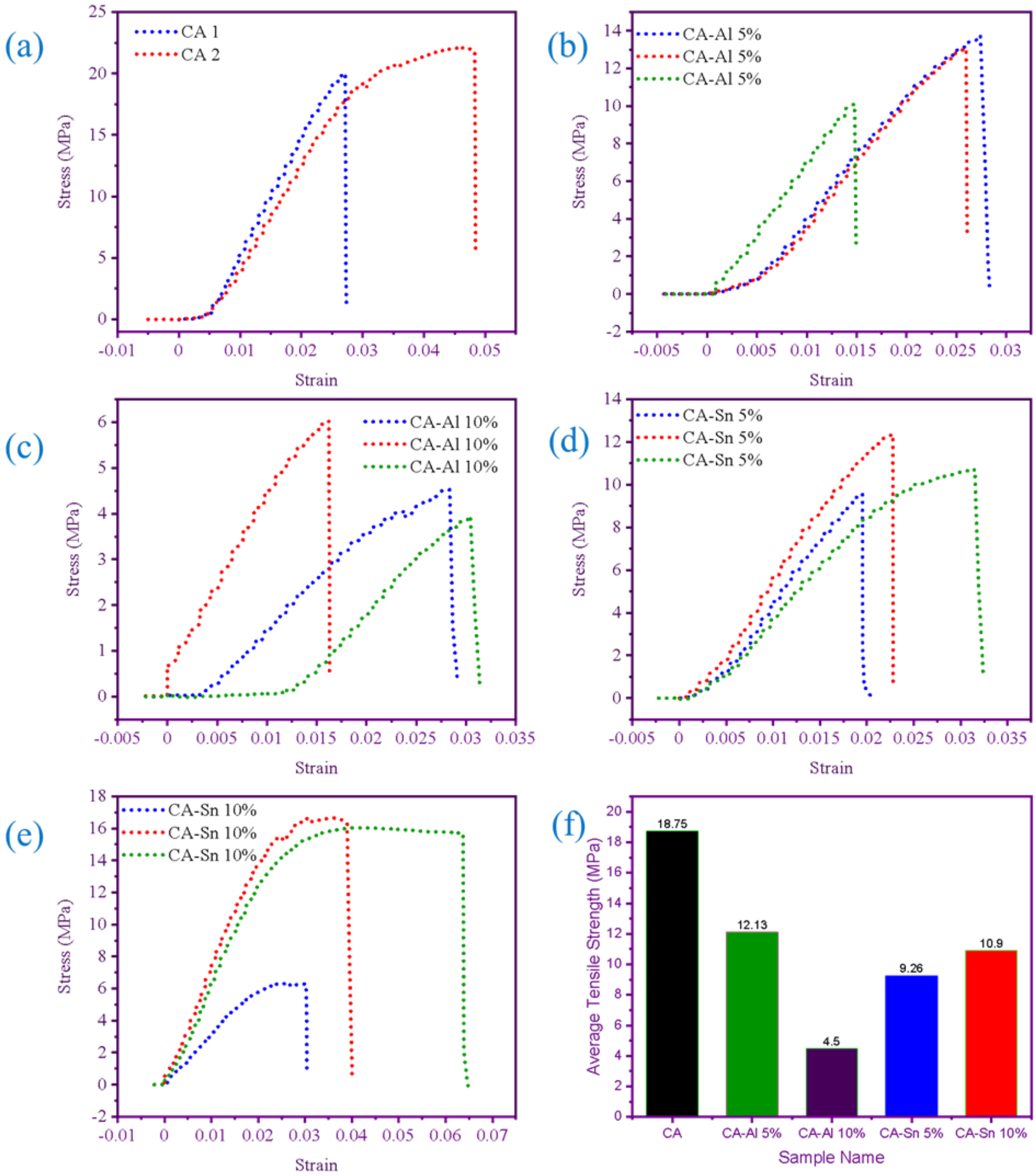


Figure 5.11 Stress-Strain Curves of (a) CA (b) CA-Al 5% (c) CA-Al 10% (d) CA-Sn 5% (e) CA-Sn 10% (f) Average Tensile strength comparison of all samples

Conclusion

New regulations in Energy conservation have made a steep increase in the demand for heat-reflecting called as Low-E films on the architectural glass as well as coating systems and window films. To deposit optical thin film systems modern ways are used nowadays, the fastest modification of these coating and films base on several parameter cost, economically, environmentally, advance features, and lifetime durability the key of **this research were low emissivity polymeric composite films based on Commercial metal dust into Cellulose acetate for the first time in the low e films industry**. A huge variety of different designs and type already exists, and optimization work nowadays aims at high light transmittance, stability opposes to environmental attacks. Whereas considerable progress has been made already in achieving these goals. Ideal low-E coating and films are still a dream. Real systems either unsatisfactory in meeting all the requirements expressed by the glass industry or are much too expensive for the architectural market. So, the commercial used at low-cost films that can be affordable for everyone in the Low e film having Low e characteristics we accomplished in this research. An emissivity value determines of fabricated films around 3.2 $W/m^2 \cdot K$ to 3.3 $W/m^2 \cdot K$ for Al/CA and 3.1 $W/m^2 .K$ to 3.8 $W/m^2 .K$ for Sn/CA whereas solar radiations reflectance about 80% in infrared region. The characterized low emissivity films incorporated by metal dust are tinted, uniform, and flexible films exhibit excellent performance for window application.

Future Recommendations

Following further work and improvement can be done for development of present results. Significantly, the pace for improvements remains, as in this research future recommendation is to add a self-cleaning property, frosting effects, tinting and privacy film also recommended to work on mechanical strength properties of these film that keeps everyone interested or involved in this field busy for the upcoming few years. Next basic recommendations of these films are mentioned below

1. **Large Area Casting.**

For the commercialization of these films, they must be deposited on large area samples.

2. **Low emissive Properties of other metals**

Other metals show promising emissive ability such as Silver, Gold which need to be deposited for better comparison and more effective results as they are expensive but have low emissivity than Al & Sn.

3. **Mechanical Testing of Thin Film**

The thin films need to be tested for their mechanical strength against abrasiveness. This can be done by the following tests:

- ✓ Scotch tape peel-off test
- ✓ Weather resistance tests
- ✓ Tinting and frosting effect

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