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OPTICAL PROPERTIES AND MATHEMATICAL MODEL FOR WATER BASED ALUMINA & TITANIA NANOFUIDS
 MOHAMMAD SAJID HOSSAIN [FACULTY OF ENGINEERING UNIVERSITY OF MALAYA KUALA LUMPUR 2013](#) OPTICAL PROPERTIES AND MATHEMATICAL MODEL FOR WATER BASED ALUMINA & TITANIA NANOFUIDS MOHAMMAD SAJID HOSSAIN [DISSERTATION SUBMITTED IN FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF MASTER OF ENGINEERING SCIENCE FACULTY OF ENGINEERING UNIVERSITY OF MALAYA KUALA LUMPUR 2013](#) UNIVERSITI MALAYA [ORIGINAL LITERARY WORK DECLARATION Name of Candidate:](#) Mohammad Sajid Hossain ([I.C/Passport No.:](#) AB 0433676) [Registration/Matric No.:](#) KGA110078 [Name of Degree:](#) Master of Engineering Science [Title of Project Paper/Research Report/Dissertation/Thesis \("This Work"\):](#) Optical Properties and Mathematical Model for Water Based Alumina & Titania Nanofluids Field of Study: Advance Materials/Nanomaterials [I do solemnly and sincerely declare that: \(1\) I am the sole author/writer of this Work; \(2\) This Work is original; \(3\) Any use of any work in which copyright exists was done by way of fair dealing and for permitted purposes and any excerpt or extract from, or reference to or reproduction of any copyright work has been disclosed expressly and sufficiently and the title of the Work and its authorship have been acknowledged in this Work; \(4\) I do not have any actual knowledge nor do I ought reasonably to know that the making of this work constitutes an infringement of any copyright work; \(5\) I hereby assign all and every rights in the copyright to this Work to the University of Malaya \("UM"\), who henceforth shall be owner of the copyright in this Work and that any reproduction or use in any form or by any means whatsoever is prohibited without the written consent of UM having been first had and obtained; \(6\) I am fully aware that if in the course of making this Work I have infringed any copyright whether intentionally or otherwise, I may be subject to legal action or any other action as may be determined by UM. Candidate's Signature Subscribed and solemnly declared before, Witness's Signature Name: Designation: Date Date ii](#)

ABSTRACT The purpose of [this research is](#) to experimentally investigate optical behaviour of Alumina and Titania nanofluids. Effects of aggregation on optical properties along with development of a mathematical model for extinction coefficient have also been done. In this dissertation, Classical theories such as, Rayleigh, Quasi Crystalline, Maxwell-Garnett and Lambert-Beer's approaches are used for analytical analysis. Experiment is conducted for 0.03, 0.05 and 0.08 %v/v concentrations at different time intervals. Outcome of the dependencies are then combined together to obtain a realistic mathematical model for measurement of extinction coefficient. Results of the study show that Alumina nanofluids are very stable for 0.03 %v/v concentration comparative to Titania nanofluids in a basefluid of pH 4 at room temperature. However, extinction coefficient and refractive index of Titania nanofluids are found higher than that of Alumina nanofluids in visible region of light for all concentrations. At the first hour, in the visible region (400-700 nm), extinction coefficient of water (basefluid) was enhanced by Alumina by averagely 5.5, 9.5 and 18.7 times for 0.03, 0.05 and 0.08 %v/v concentrations respectively. On the other hand, at the first hour in the visible region (400-700 nm), extinction coefficient of water (basefluid) was enhanced by Titania by averagely 89.5, 107 and 116 times for 0.03, 0.05 and 0.08 %v/v concentrations respectively. Reductions in extinction coefficients at different stages after preparation are found very low (around 10%) for Titania nanofluids comparative to Alumina nanofluids (around 30%). It is also found that the proposed model gives accuracy more than 65% for Alumina in the range of 350-1100 nm for up to 0.08 %v/v concentration. On the other hand, it can give accuracy more than 72% for Titania nanofluids in the range of 650-1100 nm wavelength for up to 0.05 %v/v concentration. iii As a conclusion, Alumina is found good in the sense of stability. Although optical enhancement of it is lower than that of the Titania, Titania is less stable. Titania may perform as a good solar irradiation absorber if it can be stabilized properly. The proposed model is also a good achievement, since it can give more than 65% accuracy. It is worth mentioning that present available models hardly provide up to 32% accuracy for Titania and the value is even less for Alumina nanofluids. Results of this work will be very helpful in analysing direct absorption solar collectors using Alumina and Titania nanofluids. Other nanofluids can also be investigated for different concentrations and sizes to enrich the data for practical usage and development of new solar energy harvesting technologies, such as direct absorbing solar collectors. iv **ABSTRAK** Tujuan penyelidikan ini adalah untuk menyiasat tingkah laku optik bagi Alumina dan Titania cecair nano. Kesan pengagregatan pada sifat optik bersama-sama dengan pembangunan model matematik bagi pekali kepupusan juga telah dilakukan. Dalam disertasi ini, teori-teori klasik seperti, Rayleigh, Quasi kristal, Maxwell-Garnett dan pendekatan Lambert- Beer digunakan untuk analisis analitikal. Eksperimen dijalankan untuk 0.03, 0.05 dan 0.08 %v/v kepekatan pada jangka masa yang berbeza. Hasil kebergantungan kemudiannya akan digabungkan bersama-sama untuk mendapatkan satu model matematik realistik untuk mengukur pekali kepupusan. Keputusan kajian menunjukkan bahawa cecair nano Alumina sangat stabil untuk 0.03% v/v kepekatan berbanding Titania cecair nano dalam cecair asas dengan pH 4 pada suhu bilik. Walau bagaimanapun, pekali kepupusan dan indeks biasan cecair nano Titania didapati lebih tinggi daripada cecair nano Alumina di kawasan cahaya tampak untuk semua kepekatan. Pada jam pertama, di dalam julat cahaya tampak (400-700nm), pekali kepupusan air (cecair asas) telah ditingkatkan keseluruhannya oleh Alumina dengan 5.5, 9.5 dan 18.7 kali bagi kepekatan 0.03, 0.05 dan 0.08 %v/v. Sebaliknya, pada jam pertama di rantau dilihat (400-700nm), pekali kepupusan air (cecair asas) telah ditingkatkan dengan Titania keseluruhannya 89.5, 107 dan 116 kali untuk kepekatan 0.03, 0.05 dan 0.08 %v/v. Pengurangan pekali kepupusan pada peringkat yang berbeza selepas penyediaan didapati sangat rendah (kira-kira 10%) untuk Titania cacair nano berbanding cecair nano Alumina (sekitar 30%). Selain daripada itu, juga didapati bahawa model yang dicadangkan memberi ketepatan lebih daripada 65% untuk Alumina dalam julat 350-1100 nm sehingga kepekatan 0.08 %v/v. Sebaliknya, ia boleh memberikan ketepatan lebih daripada 72 % untuk cecair nano Titania dalam julat panjang gelombang 650-1100 nm sehingga kepekatan 0.05 % v/v. v **Kesimpulannya**, Alumina didapati bagus dari segi kestabilan. Walaupun peningkatan optiknya adalah lebih rendah daripada Titania tetapi Titania telah dibuktikan sebagai kurang stabil. Titania mungkin boleh menjadi penyerap sinaran solar yang baik jika ia boleh distabilkan dengan betul. Model yang dicadangkan adalah juga satu pencapaian yang baik, kerana ia boleh memberikan lebih daripada 65% ketepatan. Ia adalah bernilai menyebut bahawa model ini didapati jarang sekali memberikan ketepatan 32% untuk Titania dan nilai tersebut adalah lebih kurang untuk cecair nano Alumina. Hasil kerja ini akan dapat membantu dalam menganalisis penyerapan pengumpul suria dengan menggunakan Alumina dan cecair nano Titania. Cecair nano yang lain juga boleh diselidik kerana kepekatan dan saiz yang berbeza untuk memperkayakan data untuk kegunaan praktikal dan pembangunan teknologi menuai tenaga solar baru, seperti menyerap pengumpul suria secara langsung. vi **ACKNOWLEDGEMENTS** First of all, I would like to express my [gratitude to](#) the [almighty](#) Allah who has created the whole universe, taught by pen and taught man that he knew not. [I would like to extend my](#) gratefulness [to my supervisors Professor](#) Dr. Saidur Rahman [and Dr.](#) Mohd Faizul bin Mohd Sabri for their [continuous support of my](#) M.Eng.Sc. [study and research](#), for their [patience, enthusiasm, motivation and immense knowledge](#). I would also [like to](#) acknowledge Professor Dr. Faisal Rafiq bin Mohammad Adikan [for his guidance. I am grateful to Dr.](#) Mahendra Varman and Dr. Ang Bee Chin for providing

laboratory support. I am thankful to Mr. Mohammad Said bin Sakat, Mrs. Azira Idris, Ms. Norhaya Abdur Rahim for their technical support during experiment. I am also grateful to Mr. Zafar Said and Ms. Nurul Fizan Sidek [for their support and encouragement](#) throughout [the study. I am](#) certainly indebted [to my](#) parents, Mohammad Jalal Uddin and Shahida Khatun, my brothers, Mohammad Nizam Uddin and Mohammad Sabbir Hossain. Their love, motivation and support always inspire me at every stage of my life. It would be impossible to complete this dissertation without their support and encouragement. Finally, I am very grateful to [the Ministry of Higher Education, Malaysia](#), and UPGP [for providing financial support](#) to me through the project FP019-2011A under the Fundamental Research Grant Scheme (FRGS). Last but not the least, I do appreciate the privileges and opportunities offered by the University of Malaya and thankful to its each and every staff for their direct or indirect cooperation. [vii TABLE OF CONTENTS ORIGINAL LITERARY WORK DECLARATION ii ABSTRACT iii ABSTRAK v ACKNOWLEDGEMENTS vii TABLE OF CONTENTS viii LIST OF FIGURES xi LIST OF TABLES xiv LIST OF SYMBOLS AND ABBREVIATIONS xv LIST OF APPENDICES xix CHAPTER 1: INTRODUCTION 1 1.1 Introduction](#)

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ATM BRIC Ccc DASCs DLS DLVO EG FESEM GHG NP NF OECD PG pzc STM SEM TEM UV XRD A B(h) D Dh Dt Fb Fr Fg LIST OF SYMBOLS AND ABBREVIATIONS Atomic force Microscopy Brazil, Russia, India and China Critical coagulation concentration Direct Absorption Solar Collectors Dynamic Light Scattering Derjaguin-Landau-Verwey-Overbeek Ethylene Glycol Field Emission Surface electron Microscopy Green House Gas Nanoparticle Nanofluid Organisation for Economic Co-operation and Development Polyethylene Glycol Point of Zero Charge Scanning-Tunnelling Microscopy Surface Electron Microscopy Transmission Electron Microscopy Ultra Violet X-Ray Diffraction Hamaker Constant Hydrodynamic interaction factor Particle diameter Hydrodynamic diameter Translational diffusion coefficient Buoyancy force Resistance force Gravity force xv I Intensity of light Io Intensity of incident light J Ion Concentration M Molar concentration of ions N Avogadro's number Nint Particle number in a single aggregate QCA Quasi Crystalline Approach Qext Extinction efficiency Qabs Absorption efficiency Qscat Scattering efficiency RA Rayleigh Approach Ra Radius of gyration Re Equivalent Radius RH Hydrodynamic radius T Thermodynamic temperature Tr Transmittance V Energy Vmax Net maximum energy barrier VT Total energy Vr Repulsive force between the particles Va Attractive force between the particles W Stability ratio d Surface distance between particles df Fractal dimension e Electron charge h Interparticle distance k Extinction index kB Boltzmann's constant xvi m Complex refractive index ratio of particle to medium ma Mass of the particles in a single aggregate mp Mass of a single particle n Real part of complex refractive index n _r np Radius of particles t Time tp Aggregation time constant v/v Volume fraction z Valence of ions α Size parameter δ Thickness of medium ε Dielectric constant εr Relative dielectric constant of the solvent εo Dielectric constant of free space η Dynamic viscosity κ Debye parameter λ Wavelength μ Viscosity of the liquid ν Sedimentation velocity ρ Mass density ρext Extinction coefficient ρabs Absorption coefficient ρscat Scattering coefficient φ Volume Fraction φp Volume fraction of particles φint Volume fraction of the particles in the aggregates φ Volume fraction of the aggregates in the entire fluid ψ Zeta potential xviii eff Effective medium ' Real part " Imaginary part xviii LIST OF APPENDICES Table A.1: Viscosity of water at different temperatures.....	98	Table A.2: Experimental transmittance of Alumina nanofluids at different time periods after preparation.....	99
Table A.3: Experimental transmittance of Titania nanofluids at different time periods after preparation.....	99	CHAPTER 1: INTRODUCTION 1.1 Introduction High global energy demand rate and its effect on environment can be coped by combining natural renewable and sustainable resources with fossil fuels. Alarming increment rate of GHG which is calculated as 3/4th of total GHG emission only within last 20 years can be minimized using the natural energy resources such as, solar radiation, wind and water. These resources can also provide secure energy supply to maintain rapid economic growth as these resources are adequately available in nature and do not have dependency on any type of geopolitical or economical breakdown. Harvesting the enormous solar energy to meet the demand is the most popular option. Although different types of solar thermal collectors are presently in use, regular design and development is continuing to integrate new inventions or technologies to the systems. A new kind of heat transfer fluid has been developed which is called nanofluid. It shows unique thermo-mechanical properties. Currently it is found that nanofluids are good solar radiation absorber also. Nanofluids have been investigated in different types of solar collectors and volumetric solar collectors are found more efficient than the conventional flat plate solar collectors. Although solar energy offers great opportunity to overcome the worst effects of	100

conventional fossil fuels, discovery of methods for conversion and harvesting such green energy remains as the most important challenge. Nanofluids, having remarkable enhancement in heat transfer capacity, are attracting researchers to implement them in solar cultivating systems. Nanoparticles are being found very good electromagnetic wave (light) absorber within UV-Visible range where 85% of solar energy is dissolved. On the other hand, conventional base fluids absorb the energy laid within infrared region which consist of 15% of solar energy. The change in properties of nano-scale materials compared to its bulk might be for their surface atoms. Amount of 1 surface atoms in nano-scale materials drastically increases more than the amount in bulk, e.g. Fe cube of 10nm each edge has 10-5% atoms as surface atoms whereas a cube of 1 nm each edge of same material has 100% atoms as its surface atoms (Link & El-Sayed, 2000). 2/Water Nanofluid", Modern Applied Science, 2013.">[Surface to volume](#) ratio 2/Water Nanofluid", Modern Applied Science, 2013.">[of nano-scale materials](#) 2/Water Nanofluid", Modern Applied Science, 2013.">[is](#) also 2/Water Nanofluid", Modern Applied Science, 2013.">[very](#) high, which is making them a good carrier of energy. Combination of nanoparticles and base fluid as colloidal working medium is expected to have a high impact on efficiency of solar cultivating systems. The fundamental optical properties (absorption, scattering and extinction coefficients, refractive index, etc.) of nanoparticles are being investigated by researchers of different fields around the world. However, very few works have been done on optical properties of nanofluids. Recent literatures, such as (He et al., 2013), have reported good thermal and optical enhancement by nanofluids in comparison with other basefluids. Moreover, Alumina and Titania nanoparticles are reported enhancing thermal conductivity of basefluids (Yiamsawasd [et al.](#), 2012; [Murshed et al.](#), 2008; Wang [et al.](#) 2007). Besides, these nanoparticles are inexpensive and commercially available to be considered for large scale engineering applications. In addition, optical properties of nanoparticles also depend on the characteristics of surrounding medium (Mahmoud et al., 2012). Hence, the properties of nanoparticles are not exactly same as nanofluids. Furthermore, the reported data are not enough for practical implementation or analysis of the Direct Absorption Solar Collectors (DASCs) as they are not free from controversy statements and limitations. Therefore, it is essential to investigate optical properties of nanofluids.

1.2 Statement of Problem Controversy statements and limitations of the available models for optical properties estimation of nanofluids based on Rayleigh, Maxwell-Garnett and Mie & Gans approaches are the thrust for this present work. The study intends to investigate optical behaviour of nanofluids for the purpose of their implementation in direct absorption solar collectors. Specifically, the study seeks to answer: a. How does growth of aggregates and concentration affect optical properties of nanofluids? b. Are all the nanofluids having high extinction coefficient applicable for direct absorption solar collectors? c. Is there any mathematical correlation to estimate optical behaviour of the nanofluids which can overcome the limitations of recent fundamental theory based models? 1.3 Objectives of the Study To be able to solve the stated problems and to answer the research questions, the [objectives of the study are](#) considered [as follows](#): 1) To study stability of Alumina and Titania nanofluids. 2) To investigate the optical properties of nanofluids both analytically and experimentally (extinction, scattering, absorption coefficients and refractive index of Alumina and Titania nanofluids). 3) To develop a best fitted mathematical model to correlate size and concentration of nanoparticles with extinction coefficient of Alumina and Titania nanofluids. 1.4 Boundary of the Study Extinction coefficients and refractive index of Alumina and Titania nanofluids were experimentally investigated. Analytical analyses of absorption, scattering and extinction coefficients, and refractive indexes have been done. However, it had not been possible to measure scattering coefficient experimentally due to high cost of testing accessories and unavailability of such accessories in Malaysian universities and institutes. Moreover, measurement of refractive index for the whole range of solar spectrum could not be done as well due to inaccessibility of wide range of laser. As a result, it has been agreed to continue the investigation with available equipment. Therefore, extinction coefficients 3 were experimentally measured for a wavelength range of 190-1100 nm and refractive index was measured only at 589.3 nm wavelength. Other supporting information regarding scattering and absorption phenomena has been collected through extensive study of related subjects. However, a correlation for the nanofluids to estimate extinction coefficients has been proposed which is able to approximate the values of extinction coefficients at low concentrations (≤ 0.08 %v/v). A [flow chart of the](#) whole thesis [is](#) presented [in Figure 1. 1](#) as a summary. 1 [.5 Outline of the Dissertation](#) This dissertation consists of five chapters. Brief description of each chapter has been presented as follows: CHAPTER 1: This is the introductory chapter of the dissertation that shows an overview of the present and future effects of fossil fuel on environment, economy and development. It also highlights about necessity of development of technology for harvesting solar energy as an alternative energy source. Lastly, objectives and limitations of the study are illustrated. CHAPTER 2: Required information related to the study has been extensively reviewed in this chapter. Information regarding nanofluids, its characterization and preparation has been described first. Secondly, a focus on stability of nanofluids and related parameters to it has been given followed by optical properties. In the sections of optical properties, available data on optical properties, mathematical models and their overview have been illustrated. CHAPTER 3: Design, method and procedures have been described in this chapter. Design and procedure of Alumina and Titania nanofluids are described in the first section. [In the second section of this chapter](#), method of stability estimation has been demonstrated followed by the section describing the procedure to measure optical properties. Lastly, method for development of a correlation has been illustrated. CHAPTER 4: This is the chapter for presenting results and interpretations. Experimental and analytically estimated values of the optical properties have been compared and presented. Effect of aggregation on optical properties has also been discovered. Lastly, a correlation for estimating optical properties has been proposed. CHAPTER 5: All the presented results in Chapter 4 have been explained and discussed in this chapter. CHAPTER 6: In this concluding chapter, outcomes of the dissertation have been summarized and some recommendation for possible future work have been mentioned. [Figure 1.1: Flow chart of the research](#) work CHAPTER 2: LITERATURE REVIEW [2.1](#) Introduction [Since Choi](#) generated [the novel concept of nanofluids in the spring of 1993, scientists and engineers in the rapidly growing](#) nanofluid [community have made scientific](#) advancement [not only in discovering](#) unique [thermal properties, but also in proposing new mechanisms behind enhanced thermal properties of nanofluids](#) (Das, 2008). Researchers are [developing unconventional models of nanofluids and identifying unusual opportunities to develop next-generation](#) heat transfer fluids [such as smart coolants for computers, safe coolants for nuclear reactors](#) and heat transfer medium for solar collectors. Nanofluids are also reported for high light absorption capacity. Consequently, [the research topic of nanofluids has been receiving increased attention worldwide](#). Rapidly increasing number of publications on nanofluids is the strong evidence on the growth of work in this [area. The main objective of this chapter is to draw up a big picture of the small world of nanofluids through a](#) comprehensive [review of](#) the concept of nanofluids, its preparation and stability, its mathematical models for optical properties, and potential applications and benefits of nanofluids. Study has been conducted through a sound collection of related [PhD and Master thesis, journal articles](#), reports, [conference](#) articles, internet sources [and](#) books. It is noteworthy that 80-90% of the journal articles are collected from the most pertinent and prestigious peer reviewed international journals, such as Renewable Energy, Solar energy, Renewable and Sustainable Energy, Physical Chemistry, Nanoparticles research, Nanomaterials, Applied Physics, Renewable & Sustainable Energy Reviews, Heat and Mass Transfer, etc. Moreover, substantial amount of relevant information has been gathered through personal contact with the key researchers around the world. 2.2 Necessity of Alternative Energy Sources An alternative energy source is the main concern of this age. Estimated energy demand of 60% from 2002 to 2030, i.e. 1.7% per year (Solangi et al., 2011) as an addition becomes a great problem to handle with. However, energy is considered as the lifeblood, its demand must be fulfilled to continue economic progress and development. According to the World

Economic Forum's "Energy Vision Update 2012" report, in 2010, energy consumption rate of OECD countries increased by 3.5% whereas for BRIC countries the rate is increased by 7.5% (12% up from 2009) results energy consumption of 5.6 billion tons of coal equivalent. It has also been estimated that such energy consumption in only China will result to 4 billion metric tons of coal equivalent which is the half of total world's demand (Yergin & Gross, 2012). As a result, this condition leads to mining fossil fuel resources at an increasing rate (Asnaghi & Ladjevardi, 2012). Geopolitical conflicts are also continuing to impact on fossil fuel price, the price hits \$126.65 per barrel in 2011 (Yergin & Gross, 2012). It may become much higher if the issues with the fuel supplying countries are not resolved. Moreover, the impact of energy consumption is not only related to the economic growth, it has a considerably high impact on the global environment also. Today's greenhouse effect and global warming are the result of not other than the increased emission of carbon dioxide in environment by the fossil fuels. China, among the BRIC countries, is alone producing a huge amount of GHG which reached 8 billion metric ton in 2011 (approximately 24% of total emission) (Yergin & Gross, 2012). Therefore, increasing price of fossil fuel and environment pollution rate have left no other way but finding alternative sources of energy.

2.3 Recent Works on Solar Thermal Energy Harvesting with Nanofluids Enhancement in efficiency of various amounts was reported by researchers for different types of solar cultivating systems with nanofluids such as, 3% and 5% (Otanicar et al., 7 2010) for DASCs with graphite (30 nm) and silver (20 nm) nanofluids relative to flat solar collector with water as working medium respectively; 5%-10% (Taylor et al., 2011) for dish solar receiver with graphite nanofluid relative to conventional fluid; 28.3% (Yousefi et al., 2012) for flat plate solar collector with Al₂O₃/H₂O nanofluids relative to conventional fluid; Ni nanofluid is also found as a good working fluid for solar collectors (Kameya & Hanamura, 2011).

2.4 Nanofluids Modern nanotechnology can produce metallic or nonmetallic particles of nanometer dimensions. Nanomaterials have unique mechanical, optical, electrical, magnetic, and thermal properties. Nanofluids are engineered by suspending nanoparticles with average sizes below 100 nm in traditional heat transfer fluids such as water, oil, and ethylene glycol. Nanofluids (nanoparticle suspensions) is the term coined by Choi and Eastman (1995) to describe this new class of nanoparticle based heat transfer fluids. Nanofluid technology, a new interdisciplinary field of great importance where nanoscience, nanotechnology, and thermal engineering meet, has developed largely over the past decade. The goal of nanofluids is to achieve the highest possible thermal properties at the smallest possible concentrations (preferably <1% by volume) by uniform dispersion and stable suspension of nanoparticles (preferably <10 nm) in host fluids. From a general perspective, a two-phase colloidal system can be classified in terms of a dispersed phase and a dispersion medium. The dispersed phase and dispersion medium can be any one of the three phases (i.e., gas, liquid, or solid) except that the first category (i.e., gas in gas) is unknown. From this, a solid nanoparticle dispersed in an amorphous solid may be considered as a colloidal system and consequently, a nanofluid. In our descriptions, fluids will be liquids at ordinary conditions of temperature and pressure, and for that reason, supercritical fluids and gases as the dispersion phase are not considered.

Domain of nanofluids (colloids) lays between Helmholtz-Smoluchowski limit and Huckle limit. Figure 2.1 presents the domain in terms of radius of particles and Debye parameter. Figure 2.1: Domain of aqueous colloidal system in terms of radius of particles and Debye parameter. (Source: Hiemenz & Rajagopalan, 1997)

2.4.1 Characterization of Nanoparticles There are different characterization processes for nanomaterials, such as X-Ray Diffraction (XRD), Dynamic Light Scattering (DLS), Optical Spectroscopy, Raman Spectroscopy, Surface Electron Microscopy (SEM), Field Emission Surface electron Microscopy (FESEM), Transmission Electron Microscopy (TEM), Scanning-Tunnelling Microscopy (STM), Atomic force Microscopy (AFM), etc (Alyamani & Lemine, 2012; Goldstein et al., 2003; Herrera & Sakulchaicharoen, 2009; Xu, 2001). All the processes are important for relevant specific perspective. In our case, the main concern is the size and distribution of nanoparticles that can be characterized by using DLS and SEM/FESEM/TEM. Therefore, the discussion about the characterization will be limited to those selected processes only.

2.4.1.1 Dynamic Light Scattering Approach Dynamic Light Scattering is also known as Photon Correlation Spectroscopy. This is one of the popular method of measuring particle size. Illumination of monochromatic light on a colloidal solution having spherical particles in Brownian motion causes Doppler shift when light hits particles. This shift changes the wavelength of incoming light which change is used to measure size distribution of particles by calculating diffusion coefficient and with the help of auto correlation facility (Xu, 2001). Brownian motion is modelled by the Stokes-Einstein equation. The equation is given below in the form of most often used for particle size analysis. The Stokes-Einstein relation connects diffusion coefficient measured by dynamic light scattering to particle size where, D_h is the hydrodynamic diameter, D_t is the translational diffusion coefficient, k_B is Boltzmann's constant, T is thermodynamic temperature, η is dynamic viscosity (Pabst & Gregorova, 2007). $D_h = \frac{k_B T}{3\pi\eta D_t}$ (2.1) The equation does serve as important reminder about a few points. The first is that sample temperature is important, as it appears directly in the equation. Temperature is even more important due to the viscosity term since viscosity is a stiff function of temperature. Finally, and most importantly, it reminds the analyst that the particle size determined by dynamic light scattering is the hydrodynamic size. That is, the determined particle size is the size of a sphere that diffuses the way as particle (Dynamic Light Scattering, 2012).

2.4.1.2 Electron Microscopy Approach Electron Microscopy operates on the same basic principles as the light microscopy but it uses electrons instead of light. The resolution of the optical microscopy is restricted by the wavelength of visible light, which thus prevents atomic-scale imaging. On the other hand, an energetic electron has a wavelength of much less than 1 Å, so that an enormous improvement in resolution can be achieved by using a beam of fast electrons for imaging (Alyamani & Lemine, 2012). There are mainly two types of electron microscopes: Scanning Electron Microscopes (SEM), and Transmission Electron Microscope (TEM). These types of microscopes detect the emitted or transmitted electrons from the surface of the sample for imaging. The accelerated voltage is ranging from 10 kV to 40 kV for the SEM and greater than/equal 100 kV for the TEM. Since the electrons are easily scattered in air all electron microscopes should operate under a high vacuum (Alyamani & Lemine, 2012).

2.4.1.2.(a) Transmission Electron Microscopy (TEM) Transmission electron microscopy is a microscopy technique where an electron beam is transmitted through an ultra-thin specimen. An image is formed from the interaction of the electrons transmitted through the specimen. A suitable combination of (magnetic) electron lenses is required, both for focusing the electron beam onto the object and also for providing an enlarged image. Million times magnifications is possible by the microscopy to visualize nano-scale materials on final viewing screen or medium (Smith, 2007). Transmission electron microscopy has emerged as a very powerful tool for detail characterization of nanomaterials. Along with imaging of nanomaterials, it can give structural (atomic arrangement), composition and geometric information of the nanostructures. Besides, it can reveal the crystallographic defects with the help of high resolution imaging (HREM) mode (Neogy et al., 2006). In spite of all these advantages, TEM imaging still presents a series of challenges. For example, overlapping of image is a typical problem during observation. When it occurs, the surrounding matrix usually tends to mask the supported nanoparticles. In some special cases, however, the existence of an epitaxial relationship between the nanoparticles and their support can be used to obtain size and shape information (Smith, 2007). Moreover, nanoparticles can be susceptible to damage under the electron beam irradiation conditions normally used for high-resolution imaging (Herrera & Sakulchaicharoen, 2009). In TEM the transmitted electrons are detected, and in this case the specimen thickness is important and typically should not exceed 150 nm (Alyamani & Lemine, 2012).

2.4.1.2.(b) Scanning Electron Microscopy (SEM) Scanning electron microscopy is a microscopy technique where scattered electrons are collected to generate image of the specimen. Thermionic emitter is used for emitting electron over the specimen surface. The thickness of the specimen in this case is not important. In addition, the samples to be tested have to be electrically conductive; otherwise they would be overcharged with

[electrons. However, they can be coated with a conductive layer of metal or carbon.](#) SEM mainly used for topographical images. However, elemental analysis can also be carried out to obtain compositional information. It [can yield valuable information](#) regarding [the purity of a nanoparticle sample as well as an insight on their degree of aggregation](#) (Goldstein et al., 2003). Moreover, when nanoparticles are part of secondary and tertiary nanostructures, SEM becomes a valuable tool to assess their location (Debe, 2010). [SEM is, to a certain extent, a limited tool to characterize nanoparticles. The main problem with the application of SEM to nanoparticle characterization analysis is that sometimes it is not possible to clearly differentiate the nanoparticles from the substrate.](#) Problems become even more exacerbated when the nanoparticles under study have tendency to adhere strongly to each other, forming agglomerates. In contrast to TEM, SEM cannot resolve the internal structure of these domains. 12.2.4.1.2.(c) [Field Emission Scanning Electron Microscopy](#) [The FEM is a state-of-the-art electron microscopy. The new field-emitter technology and advanced Gemini column allow for ultra-high resolution electron imaging.](#) It uses an electron gun for providing a huge [and stable current in a small beam.](#) [There are two types of electron emitter: thermionic emitter and field emitter.](#) Field [emitter is](#) used in FESEM and it is the main difference between this and SEM. [Thermionic sources have relatively low brightness, evaporation of cathode material and thermal drift during operation.](#) On the other hand, [Field Emission is the way of generating electrons that avoids these problems.](#) It is [also called a cold cathode field emitter](#) because it [does not heat the filament](#) to generate electron beam. [The emission is obtained by placing the filament in a huge electrical potential gradient. The FES is usually a wire of Tungsten \(W\) fashioned into a sharp point. The significance of the small tip radius \(~ 100 nm\) is that an electric field can be concentrated to an extreme level, becoming so big that the work function of the material is lowered and electrons can leave the cathode](#) (Alyamani & Lemine, 2012).

2.4.2 Preparation of Nanofluids Several types of methods are discovered for preparation of nanofluids. Among the methods, [two-step technique is the easiest one.](#) One [step technique and](#) some other novel techniques are also in use to prepare nanofluids. 2.4.2.1 Two-step Techniques [Two-step method is the most popular and economical method. Nanoparticles are dispersed in conventional fluids by means of external force. Two techniques are usually used by the researchers and scientists to disperse tiny particles in a fluid: Physical Technique and Chemical Technique. In physical technique, two types of stabilizing process can be considered, one is mechanical and another is ultrasonic. One way of mechanical dispersion is applying shear force to pull agglomerates apart—high shear mixing. Usually narrow passages and/or relatively high rates of flow are required to generate high shear.](#) For that purpose, [a rotor- and stator construction is used.](#) This includes a high-speed mixer, homogenizer, micro- fluidizer, Kady mill, and colloid mill. Another means of mechanical dispersion is high-impact mixing, which applies much higher energy to break the tightly bound aggregates apart or to shatter coherent solids into smaller pieces. Usually a grinding material of small particle size is used to exert an impact on the material or particles to be dispersed. Examples include attritor and ball-and-pebble mill. Ultrasonic homogenizer is also widely used in making nanofluids. Ultrasonic energy is a form of mechanical vibratory energy which propagates through a liquid medium as elastic waves. The ultrasonic interactions within a suspension could be mechanical, thermal, or chemical. The activator converts the regular-line frequency to a much higher level (e.g. 20,000 Hz), which is eventually converted into mechanical vibrations in the tips of various shapes (horns). A conventional bath-type sonicator provides less energy density than the tip-type one. The tip of the horn is immersed in a liquid in which the ultrasonic vibrations cause cavitation, which stirs the dispersion or breaks the agglomerates (Yu & Xie, 2012). Chemical methods are always applied as processing aids to the aforementioned physical methods. There are two kinds of stabilization method which are electrostatic and steric dispersion. In electrostatic dispersion, like electrostatic charges of sufficient magnitude are spread on the surfaces of suspended particles to repel one another and remain in stable suspension, rather than forming aggregates. Steric stabilization prevents the nanoparticles 14 from getting close enough to coalesce and precipitate by means of dispersants such as surfactants (an acronym for surface active agents), polymers, or polymeric surfactants. 2.4.2.2 One-step Techniques A simultaneous process of nanoparticle generation and dispersion in a specific fluid is called one-step method. This process is able to produce uniformly dispersed particles which make the nanofluid stable. Two methods are generally used under this technique: Direct Vapour method and liquid chemical method. The [single-step direct evaporation approach was developed by Akoh et al. \(1978\) and is named as the Vacuum Evaporation onto a Running Oil Substrate technique. The original idea of this method was to produce nanoparticles, but it was difficult to subsequently separate the particles from the fluids to produce dry nanoparticles. Eastman et al. \(1997\) developed a modified vacuum evaporation oil technique, in which Cu vapour is directly condensed into nanoparticles by contact with a flowing low- vapour - pressure liquid ethylene glycol.](#) The drawbacks of this [technique](#) however, are [that the use of low vapour pressure liquids are essential and only limited quantities can be produced.](#) The [vacuum-SANSS \(Submerged Arc Nanoparticle Synthesis System\) is another efficient method to prepare nanofluids using different dielectric liquids.](#) This method can produce nanofluids with desired morphological properties of nanoparticles. The [different morphologies are mainly influenced and determined by various thermal conductivity properties of the dielectric liquids. The nanoparticles prepared exhibit needle-like, polygonal, square, and circular morphological shapes. The method avoids the undesired particle aggregation fairly well](#) (Lo et al., 2005a; Lo et al., 2005b; Yu & Xie, 2012). [Various single-step chemical synthesis techniques are also used to produce nanofluids. For example, Brust et al. \(1994\) developed a technique for producing metallic nanoparticles in various solvents by the reduction of metal salts to produce colloidal 15 suspensions for a wide range of applications, including studies of thermal transport. Excellent control of size and very narrow size distributions can be obtained by using such methods](#) (Kebllinski et al., 2005).

2.5 Stability of Nanofluids Agglomeration or coagulation of nanoparticles not only results sedimentation or clogging but also affects thermal and optical properties. The ability of resisting aggregation or coagulation of particles in a colloidal dispersion is called colloidal stability. The stability is described as kinetic and thermodynamic in nature. Kinetic stability is defined as a consequence of [force barrier against collision between particles and possible coagulation subsequently. Thermodynamic stability,](#) on [the](#) other hand, focuses on the state of equilibrium. Lyophobic colloids are thermodynamically unstable but kinetically may remain stable (Hiemenz & Rajagopalan, 1997). Since we are dealing with lyophobic colloids the term "stability" afterwards will only mean kinetic stability. Stability relate terminology are described in the following subsections. 2.5.1 Aggregation The coarsening process [of a thermodynamically unstable dispersion is called coalescence or aggregation. Coalescence](#) refers to the [process](#) where [two or more particles fuse together to form](#) large one whereas aggregation refers to the process where particles attach together to form a bunch of particles (aggregates). The term "coagulation" is also used to describe aggregation. In many cases dispersed phase remains as aggregates rather than single particle. Figure 2.2 shows the aggregation steps in a colloidal solution. Firstly, particles move to each other and then attached to each other to form aggregates. Open structure of aggregates represents fractal nature. The pattern of aggregates are indexed by Fractal Dimension which is a unit less number. The value of d_f varies between 1.57 to 2.5 (Brasil [et al., 2001](#); Prasher [et al., 2006](#); Van Saarloos, 1987; Wentzel [et al., 2003](#)); lower value [signifies diffusion-limited cluster-cluster aggregation \(DLCCA\)](#) caused for low repulsive force and higher value [signifies reaction-limited aggregation](#) caused for high repulsive force (Prasher et al., 2006). Figure 2.3 presents some structure of aggregates with corresponding fractal dimensions. APaggrtriclaetsion: Figure 2.2: A schematic of steps in aggregation process Figure 2.3: Structures of aggregates. Generated aggregates (N =150) with predefined fractal dimension A) 1.10, B) 1.70, C) 1. 9 and D) 2.76. (Source: Bedrich, 2006) Transport of particles may occur for different mechanism depending on situation but we consider only diffusion and attraction or repulsion because of inter-particle forces. Aggregation is termed

as "perikinetic aggregation" when diffusion is the transport mechanism; on the other hand it is termed as "orthokinetic aggregation" when velocity gradient is the transport mechanism. Perikinetic results rapid aggregation rate because of the Brownian motion of the particles which is unhindered by any energy barrier against contact; however, orthokinetic results slow rate of collisions due to inter-particle net force. Interconnections of aggregates create networks and form gel as concentration of particles increases as shown in Figure 2.4. Aggregates are loose and fractal in nature. Increase in concentration doesn't fill the voids present in the structure. Important and more complex region is the intermediate one. This area is very sensitive to the details of the interaction forces since the interaction and repulsion forces are comparable to each other. Both of the extreme do not allow to form closely packed structure, but intermediate region does allow densely packed structure as shown in the Figure 2.4 (Ulrich, 1990). [Figure 2.4: Relation between inter-particle forces and corresponding particles' arrangement in mono disperse colloids](#)

2.5.2 Diffusion and Sedimentation

In the absence of any external force, the composition of a single equilibrium phase is macroscopically uniform all through. The meaning is that the density is same all over the volume. However, at molecular level, there will be a fluctuation because molecules transport from lower density to relatively higher density region. This transport is called diffusion. [Figure 2.5: Sedimentation of submerged particle in a liquid medium. The action of gravity is shown on the left side and action of both gravity and viscosity is shown on the right side. On the other hand,](#) sedimentation occurs due to gravity and, noticeably faster, in a centrifuge. To see the process of sedimentation we may consider a particle of volume V and density ρ_2 which is dispersed into a liquid medium of density ρ_1 . The process can be presented as [Figure 2.5](#). F_g stands for gravity force and F_b stands for buoyancy force. If $\rho_2 > \rho_1$ particle will sediment and if $\rho_2 < \rho_1$ particle will cream. When particle moves into a fluid medium it has to overcome the friction force offered by the medium at particle surface. This force thus can be measured using viscosity of liquid. Thus, particle has to face buoyancy and friction force against gravitational force before sedimentation. Sedimentation is measured under the term "sedimentation velocity" having unit the same as velocity unit (m/sec).

2.5.3 Stability Ratio (W)

The stability of dispersion against aggregation is articulated quantitatively as stability ratio. It is defined as the ratio of the rate of diffusion controlled inter-particle collision to the rate of interaction force-controlled inter-particle collision. Large value of W means that the dispersion is slow to aggregate whereas W of order of unity describes the dispersion as rapidly aggregating. W is mathematically presented in Equation (3.8) in conjunction with the method for stability.

2.5.4 Electrostatic Stability

If the electrostatic force resulting from overlap of electric double layer of two particles becomes repulsive and can withstand the attraction force generated by Van der Waals attraction force dispersion gets stability. This type of stability is termed as electrostatic stability. [Figure 2.4](#) also presents a schematic diagram of structure of dispersions. The figure is like a phase diagram having three regions, such as attraction, repulsion and intermediate region. Inter-particle force becomes repulsive when the particles have large enough surface charges. At this extreme the Debye thickness becomes of the order of particle size or even more. Stability at this region is considered as thermodynamic stability. Although repulsion takes place, particles can organize themselves in crystalline structure even at very low concentration (0.001 or lower). Type of crystalline structure depends on concentration of particles as well as the Debye thickness. These types of structures are usually loosely packed. On the other extreme, attraction dominates the net inter-particle force resulting thermodynamic and kinetic instability. [Derjaguin-Landau-Verwey-Overbeek \(DLVO\) theory](#), named upon the scientists, is a well-known study of stability. Its development enables us to quantitatively measure kinetic stability. [Figure 2.6](#) shows the interaction energy curve between particles. Considering V_T , V and δ as total energy, energy at a certain point and distance between the particles respectively, it can be said that the attraction force will be predominate at small and large inter-particle distance, whereas double layer repulsion dominates at intermediate distance. V_{max} presents the net energy barrier, which depends upon zeta potential, Hamaker constant and Debye thickness. Two particles must have sufficient kinetic energy to overcome the barrier energy upon collision for forming aggregate. Primary and secondary minimum regions form aggregates of strong and weak bond respectively. Aggregates of secondary minimum can be broken back to separate particles but it is impossible for the aggregates of primary minimum. [Figure 2.6: Interaction energy curve between particles](#) Though electrolyte is responsible for repulsion due to double layer excess quantity of it squeezes double layer thickness and reduce repulsion force. [Figure 2.7](#) illustrates that increase in electrolyte concentration leads to compression of double layer (κ_2 increases) resulting low energy barrier, $V(2)$. DLVO theory provides a quantitative explanation of proper amount of electrolyte against coagulation. The term used for the measure is "critical coagulation concentration (ccc)" which is defined as the quantity of electrolyte at which colloids aggregate. Ccc is calculated using Schulze-Hardy rule and is expressed in mole per litre. [This rule states that it is the valance of the ion of opposite charge to the colloid that has the principal effect on the stability of the colloid.](#) [Figure 2.8](#) has been drawn for $r_p=100$ nm, $A=10^{-19}$ J, $\psi=25.7$ mV to show the character interaction/potential energy curve for different values of κ . [Figure 2.7: Total interaction energy curves \(V\(1\) and V\(2\)\) for two different electrolyte concentrations.](#) Here, ionic strength $\kappa_1 < \kappa_2$. [Figure 2.8: Potential energy curve versus particle separation distance.](#) (Source: Hiemenz & Rajagopalan, 1997)

2.5.5 Steric Stability

It is not always convenient to get electrostatic stability. For such situations, particle masking technology is used to get repulsive action. Suitable polymer is introduced in the dispersion to create a layer of polymer chains over the particle surface. When two masked particles come together overlapping of the mask layer produces repulsion through steric action of polymers. Stability obtain in this way is termed as polymer induced stability or steric stability.

2.6 Optical Properties

Light is defined as electromagnetic radiation in the wavelength range 3 nm to 30,000 nm (Xu, 2001). Upon being illuminated on surface of a matter having dielectric constant other than unity, light will be absorbed or scattered, or both, depending on the wavelength of is redirected in different directions. Scattering may simply be reflection from the surface or almost immediate emission without any change in wavelength upon a temporary absorption. Scattering, on the other hand, is also a loss of intensity of incident light caused by the obstacle molecules in the medium which force the incident light to be deviated from its straight trajectory. Absorption of light is that phenomena by which particle atoms get excited and when electrons of the atoms reach back to lower level from upper (excited) level they give up the absorbed energy in the form of thermal radiation instead of photon emission. On the other hand, net effect of scattering and absorption caused by the material is known as extinction or attenuation of light. Extinction coefficient is defined as the measure of the rate of the reduction of light through a substance (Zhang, 1990). [Figure 2.9](#) shows that the reflection of light, from the incident plane, is the combination of reflection from the cell surface and back scattering from the nanoparticles in medium 23 as well as medium molecule. The transmission of light from the rear surface of the cell is the combination of forward scattering from the nanoparticles and undisturbed light transmitted through the medium. From this analogy it can be said that the transmission and reflectance values obtained from the UV-visible spectroscopy can be considered for the measurement of the optical constants. Incident wave Internal (reflected) wave Transmitted wave Reflected wave Scattered wave Incident wave Obstacle [Figure 2.9: Analogy between scattering by a particle and reflection-transmission by a slab.](#) (Source: Bohren & Huffman, 1983)

2.6.1 Investigations on Optical Properties of Nanofluids

Extinction coefficient is dependent on size and nature of the nanoparticle, dielectric constant of the medium, temperature distribution and number of nanoparticles in a nanofluid. Different models has been developed on the basis of classical theories to describe the optical characteristics of nanofluids till date (Bohren & Huffman, 1983; Mercatelli et al., 2011b; Taylor et al., 2011). In nanofluids, absorption is for both base fluid and nanoparticles. Because of strange type of behaviours of nanoparticles in working medium it is not just simple summation of their absorption capacity for the nanofluids. It is also found that for the nanoparticles having radius

≤ 10 nm, scattering effect is not present but with the increase in size scattering becomes important (Noguez, 2005). For silver nanoparticle in water suspension, scattering increasingly becomes equal to absorption at particle size of 52 nm and it increases for further growth of the particle (Evanoff & Chumanov, 2004). It has been reported that plasmon bandwidth decreases with the increase of Au particle size up to [mean diameter of 25 nm](#) and increases for [a further increase of particle mean diameter](#) in aqueous solution (Link & El-Sayed, 1999). Ortega et al. (2008b) has found absorbance of Au/water nanofluid decreasing with increase in particle size. [The dielectric constant of surrounding medium and the presence of substrates on nanoparticle surface](#) can change the peak positions of [surface plasmon resonance of the plasmonic or metaplasmonic nanoparticles](#). The plasmonic [absorption and scattering](#) of them may also be shifted differently in different medium. Mahmood et al. (2012) has studied the effect of surrounding medium and substrates (of quartz) on Ag nano cubes of 60 nm size and found optical spectra of different peaks and ranges. He has found that the absorption and scattering band frequency depends on dielectric constant of solvent which causes band broadening; again, the [dielectric constant of the surrounding material is sensitive to the splitting of the degeneracies in the particles which occurs in the absence of a considerable shielding or polar capping of the particles](#). Plasmon bandwidth also depends on particle's size and surrounding temperature. For Al-water colloidal solution, no effect was found for the spherical particles under 20 nm diameter on absorption coefficient but the coefficient increased or the spectrum shifted upward with the increase in concentration (Saidur et al., 2012). Light absorption in fluid introduces temperature gradient, which redistributes the nanoparticles and changes the concentration of it in the fluid affecting fluid's surface tension (Lamhot et al., 2009). This rearrangement of [nanoparticles may also affect the thermal conductivity, viscosity, etc. of the fluid](#). A summary has been drawn in Table 2.1 to present other optical characterization works on nanofluids till date.

2.6.2 Models for Optical Properties of Nanofluids Different approaches are being taken by the researchers to find out a best fitted mathematical theory for describing the optical properties of nanofluids. There are few models available for estimation of optical properties. The models differ from each other due to different considerations. The models are as follows: 1) Rayleigh Approach 2) Quasi Crystalline Approximation 3) Maxwell-Garnett's Effective Medium Approach 4) Lambert Beer's Law 5) Mie and Gans theory 6) Discrete Dipole Approximation (DDA) and 7) TL and I-Scan Technique using Fresnel Approximation According to literature, single event scattering of light where particle size is very small compared to wavelength can be calculated with Rayleigh scattering. On the other hand, multiple dependent scattering occurs in nanofluids and Quasi Crystalline Approximation (QCA) is found more appropriate to calculate scattering coefficient (Prasher, 2005; Prasher & Phelan, 2005). Mercatelli et al. (2011b) used the Lambert-Beer's law to find out the optical properties. In that experiment they had used pre calibrated intralipid-20% which has high scattering and low absorption effect to find out absorption and scattering coefficients separately (Mercatelli et al., 2011b). Mie scattering procedure can be used where particle size equals wavelength and for larger particles (>10-20 times of wavelength) optical properties can be described using geometrical optics (Kuhl & Jorgensen, 1994). Nanofluid(s) SWCNHs-Water with 1.2-1.8 g/l SDS SWCNHs-Ethylene glycol Cu-Deionized water Cu-Deionized acetone Graphite-Water Al-Water TiO₂ Cu-Water Ag-Water Cu-Water Table 2.1: Works on optical characterization of nanofluids at room temperature Size of NPs (nm) Conc. 100 0.3g/l 100 0.06g/l Spherical with an n/a avg. diameter of 30 Spherical with an n/a avg. diameter of 3 Spherical with an 0.5 %v/v avg. diameter of 30. 0.1 %v/v 0.0025 %v/v <50 0.1 %v/v <50 0.5 %v/v 50 0.004 %v/v 0.004 %v/v 50 0.01 %v/v Optical characterization/ Enhancement in solar collector References efficiency Scattering $\leq 5\%$ and Absorption $\geq 95\%$ (Mercatelli et al., 2011a) Absorption coefficient [decreases from 11 cm⁻¹ to 4 cm⁻¹ for the](#) (Sani et al., 2011) wavelength range 376 nm to 1400 nm. The extinction peak was shifted from 600 nm to 400 nm wavelength (Tilaki et al., 2007) after 15 days. The extinction peak was found at around 600 nm wavelength and no change in position was observed after 15 days. Extinction coefficient decreased from 3.01 [cm⁻¹ to 2.61 cm⁻¹ with the](#) (Taylor et al., 2011) increase in wavelength (≤ 1100 nm). Extinction coefficient decreases from 2.2 [cm⁻¹ to 1.77 cm⁻¹ with the](#) increase in wavelength (≤ 1400 nm). Extinction coefficient decreases from 1.36 [cm⁻¹ to 0.93 cm⁻¹ with the](#) increase in wavelength (≤ 1100 nm). Avg. Extinction coefficient 2.34 cm⁻¹ was found within 1100 nm wavelength. Sharp decrease of extinction coefficient from 3.6 [cm⁻¹ to 0.50 cm⁻¹ with](#) an increase in wavelength up to 1300 nm was observed Extinction coefficient decreases from 1.8 [cm⁻¹ to 0.4 cm⁻¹ with](#) an increase in wavelength (<1000 nm). Extinction coefficient decreases from 1.83 [cm⁻¹ to .29 cm⁻¹ with the](#) increase in wavelength (400 nm-1300 nm). 25.3% enhancement in temperature under direct solar irradiation (He et al., 2013) 27

2.6.3 Overview of the Models Surrounding environment has a great effect on nanoparticles and their properties. A lot of efforts have been assigned for the measurement of optical properties of nanoparticles for electrical, biological and chemical uses but the measurements cannot properly predict behaviour of them in suspensions for light absorption of wider wavelength range to generate heat. Although, some models are being used for the study, they are not beyond questions. Mercatelli et al. (2012) has studied the extinction coefficient of SWCNHs-Water nanofluid and provided a spectrophotometric measurement technique to find out optical coefficient for nanofluids. They internal reflection is negligible. suited rather than Mie scattering have noted that the Rayleigh scattering function is best function, scattering is mainly for single scattering and Taylor et al. (2011) has found that their theoretical model based on the Rayleigh's scattering theory is inconsistent for the fluids other than water based graphite nanofluids. The Maxwell-Garnett effective medium approach is also found weak to predict the extinction coefficient of colloidal solution in their report. Lamhot et al. (2009) has proposed a model for optical control over surface tension of nanofluids. The model shows that the temperature and particle distribution in the medium highly controlled with the power of incident light. It is also found that change in the refractive index of nanofluids due to thermal effect is stronger than that due to concentration change of nanoparticles. As a result, increased power of incident light decreases the light absorption capacity and thus heat generation. Mercatelli et al. (2011b) have considered the fluid (nanofluid) motionless (any motion regardless convective or for pumping) and with the absence of temperature gradient to evaluate the [absorbed sunlight fraction F\(r\) as a function of Light Path Depth "r" within the fluid](#). But according to Lamhot et al. (2009), the light absorption in fluid introduces a temperature gradient which redistributes the nanoparticles and changes the concentration of it in the fluid affecting fluid's surface tension. This rearrangement of [nanoparticles may also affect the thermal conductivity, viscosity, etc. of the fluid](#). Increase in temperature of fluid by light absorption decreases the refractive index rather than changing concentration of the fluid (Lamhot et al., 2009). [A summary of the studied recent models are presented in Table 2.2](#). Researchers are still searching for an appropriate mathematical model to describe the phenomena in general.

2.7 Summary of Chapter 2 Necessity of searching alternative energy sources is discussed in conjunction with a new discovery of nanotechnology called nanofluids. Nanofluids are showing positive impression in harvesting solar thermal energy. Preparation, characterization, stability parameters and optical properties along with models of nanofluids are broadly discussed in this chapter. Two-step method is selected for preparing nanofluids. Electrostatic stability technique is considered for stabilizing the prepared nanofluids since this procedure can be applied for higher temperature compared to steric stability technique. Rayleigh, Quasi Crystalline, Maxwell-Garnett and Lambert-Beer approaches are selected for analytical analysis and new model generation because of their positive impression as presented in Table 2.2. The techniques and methods are [discussed in detail in the](#) Chapter 3. Table 2.2: Summary of [the Models of Nanofluids Model\(s\) Must to know Applicability Limitation\(s\) References](#) Rayleigh Complex ? Well fitted for water based • Not applicable for metal nanofluids as well as (Mercatelli et al., 2012; Scattering refractive index graphite nanofluids. graphite nanofluids other than water as base fluid. Saidur et al., 2012; approach of both particle • This model provides only extinction value Taylor, 2011) and base fluid. • Independent scattering is considered and thus only works

for very low concentration. Maxwell-Complex ? Refractive index values for • Extinction coefficient values for water based (Link & El-Sayed, Garnett's dielectric water based nanofluids are in nanofluids cannot be measured accurately. 2000; Taylor, 2011; effective constants of good agreement. • Neither relations provided for Taylor et al., 2011) medium both particle and ? It counts size and shape of absorption/scattering nor scattering effect is approach base fluid. nanoparticles. counted. • This model provides only extinction value. Lambert ? Applicable for homogeneous • This model provides only extinction value. (Mercatelli et al., 2011a; Beer's Law non-scattering solutions. • Scattering effect is not considered. Mercatelli et al., 2011b) • Size, shape and environmental condition are not counted. Mie and Dielectric ? Mie theory provides all the • This theory is not applicable for nanoparticles (Hartland, 2011; Gans theory of constants both particle and ? Accurately measures for of optical parameters. other than spherical and cylindrical (rod shaped). Henglein et al., 1995; base fluid. spherical particles of various sizes. Kameya & Hanamura, 2011; Link & El-Sayed, 1999, 2000) ? Gans theory is applicable for rod type nanoparticles. 30 Table 2.2 Continued Model(s) Must to know Applicability Limitation(s) References TL and I- ? Preliminary • Specific thermo-optical parameters cannot be (Jiménez-Pérez et al., scan technique using Fresnel approximat relationamong Thermo optical parameters (thermal diffusion, thermal diffusivity and dn/dT) can be obtained. obtained separately. 2011; Ortega [et al., 2008a](#); Ortega [et al., 2008b](#); Rodriguez [et al., 2007](#)) on DDA approach ? This is a numerical solution approach for arbitrary shapes of metal nanoparticles in nanofluids ? It can count the solvent and • This approach is highly dependent on the computational resource and measured electric field just near to the particle surface is inaccurate. (Mahmoud et al., 2012) substrate effect on particles of nanofluids. 31 **CHAPTER 3: METHODS AND PROCEDURE** **3.1 Introduction** This **chapter** is aimed at providing the detail description of methods and procedures to fulfil the objectives. This chapter has been divided into few subsections. Preparation of nanofluids is described in the first subsection followed by investigation of optical properties, estimation of stability and aggregation and development of mathematical model. Flowchart of the experimental and analytical steps [are presented in Figure 3.1](#). **Figure 3.1**: Flowchart of experimental and analytical analysis 3.2 Preparation of Nanofluids: Due to good thermal and optical behaviour, lack of optical data and availability in the market, Alumina and Titania nanoparticles are selected for this work. Alumina and Titania nanoparticles of 13 nm and 21 nm were selected to conduct the experiment for their availability in the market and both sizes were small enough to apply classical approach for optical analysis. The particles were purchased from Sigma-Aldrich. Al₂O₃ is amphoteric in nature; TiO₂ [is insoluble in water, hydrochloric acid](#), diluted sulphuric [acid and organic solvents](#). However, [it dissolves in hydrofluoric acid and hot concentrated sulphuric acid. It is almost insoluble in](#) alkaline [aqueous](#) solution (Kirk-Othmer, 2006). Literature says that pH value of 7-9 for Alumina (Huang et al., 2009) and pH 1-4 for Titania (Penkavova et al., 2011) give good stability. Ionic strength of base-fluid can also be adjusted tuning pH value (Min et al., 2008). Thus, hydrochloric acid solution was used to functionalize nanoparticles. The above information suggests for a weak acidic base- fluid to get aforementioned pH value. 0.0001M HCl aqueous solution was prepared to use as the base medium. A digital pH meter, [as shown in Figure 3.3](#), was used [to measure pH of the basefluid](#). Manual Mixing Ultrasonic homogenization Weighed nanoparticles in a beaker Distilled pH water+ HCl solution of 4 Prepared nanofluid **Figure 3.2: Nanofluid preparation process** [Two-step method was used to prepare nanofluids](#). One hour sonication time was considered for dispersing process. Sonication process increases the solution temperature which may have negative effect on stability. The steps of preparation is pictorially shown in the **Figure 3.2**. To overcome this risk, the experiment was conducted in a controlled environment (at 25°C) using water bath and water jacket beaker. Equation (3.1) was used to convert volume percentage to weight percentage for weighing nanoparticles (Naik & Sundar, 2011). Concentration(%v/v) = $(W_p / \rho_p) / (W_p / \rho_p + (W_{bf} / \rho_{bf}))$ (3.1) A digital weighing machine (Model: HR-250AZ from A&D Co., Japan), as [shown in Figure 3.4](#), was used [to weigh the nanoparticles](#) and an ultrasonic homogenizer (Model: FB 505 from Fisher Scientific, USA), as shown in **Figure 3.5**, was used for dispersing nanoparticles in to the basefluid. **Figure 3.3: pH Meter** **Figure 3.4: Digital Weighing Machine (HR 250 AZ)** **Figure 3.5: Ultrasonic Homogenizer (F B 505)** 3.3 Estimation of Stability of Nanofluids Colloidal solutions are considered as the non-Newtonian fluids (Hiemenz & Rajagopalan, 1997). Non Newtonian fluids are generally those fluids which do not have constant viscosity even at constant temperature and pressure. Reasons behind such behaviour of colloidal solutions are (a) inter-particle hydrodynamic interaction and (b) colloidal forces due to electrostatic effect, polymer layer effect, et c. In non-interacting dispersions concentration plays the main role but for the interacting dispersions time over which flow behaviour is observed must be considered. Colloidal forces alter with time changing the structure of the dispersion. The method described by Prasher et al. (2006) for aggregation kinetics was used [to investigate the effect of aggregation on optical properties of nanofluids as well as stability of it](#). [Volume fraction of the particles](#) can be defined as the product of [the volume fraction of the particles in the aggregates and the volume fraction of the aggregates in the entire fluid](#). Mathematically, it can be presented as Equation (3.2), where $\phi_{int} = 1$ for completely dispersed system and $\phi_a = 1$ for completely aggregated system. $\phi_p = \phi_{int} \phi_a$ (3.2) Radius of the aggregates can be presented by the radius of gyration, which can be calculated using Smoluchowski model and can be written in the form of Equation (3.3) which can be further modified as Equation (3.4), $R_a = r_p(1+t/\tau_p)^{1/df}$ (3.3) $(R_a/r_p)^{df} = (1+t/\tau_p) = N_{int}$ (3.4) ϕ_{int} can be calculated using Equation (3.5). Hydrodynamic ratio (RH/Ra) was considered 1 in this work. RH was obtained from zetasizer, where DLS method is applied to measure it. This Ra value was used to get df combining Equation (3.5) and Equation (3.7). $\phi_{int} = (R_a/r_p)^{df} - 3 = (1+t/\tau_p)^{df} - 3$ (3.5) Now, with the help of Equation (3.6) we can calculate [mass of the particles in a single aggregate](#). Considering mass of a single particle as m_p we can write Equation (3.6). Aggregation time constant was obtained from Equation (3.7), $m_a = m_p(1+t/\tau_p)$ (3.6) $\tau_p = (n_p r_p^3 W) / (k_b T \rho_p)$ (3.7) [W=1 in absence of repulsive force and hydrodynamic interaction between the nanoparticles](#) but [in presence of the repulsive force W>1](#). The threshold limit of W for dilute solution is 105 (for which $V_{total} \sim 15k_bT$) and for very concentrated solution 109 (for which $V_{total} \sim 25k_bT$) (Verwey & Overbeek, 1999). The value of W was calculated using Equation (3.8), $W = 2r_p \int_0^\infty B(h) \exp\{-(V_r + V_a)/k_bT\} / (h+2r)^2 dh$ (3.8) V_r and V_a were obtained from DLVO theory and presented by the Equations (3.9)-(3.11) for electrostatic stability. $B(h)$ was obtained using widely used formula which is presented as Equation (3.12). $V_r = 2\pi n_e r_p \Psi_2 \exp(-kh)$ (3.9) $V_r = 2\pi n_e r_p \Psi_2 \ln(1 + \exp(-kh))$ (3.10) $V_a = -(A/6)[2r_p^2 / h(h+4r) + 2r_p^2 / (h+2r)^2 + \ln(h(h+4r)/(h+2r)^2)]$ (3.11) $B(h) = 6(h/r)^2 + 13(h/r) + 2.6(h/r)^2 + 4(h/r)$ (3.12) Hamaker constant is a property of materials which is used to measure the strength of Van der Waals forces. Negative value of A presents positive Van der Waals energy (repulsive force) whereas positive value of A presents the alternative (attractive force). Equation (3.9) is valid for the nanofluids with $k r_p < 5$ whereas Equation (3.10) is valid for $k r_p > 5$. Debye parameter for water colloid without any salt can be written as Equation (3.13). But with electrolyte, the Debye parameter can be calculated using Equation (3.14) (Hiemenz & Rajagopalan, 1997). The value of Debye parameter can also be obtained from Table 3.1. (3.13) $\kappa = [1000 e N \sum z_i^2 m_i]^{1/2} \epsilon_k B T$ (3.14) i Combined Hamaker constant should be calculated for solutions. If we consider that particles of type-2 are dispersed in medium of type-1 then the combined Hamaker constant A_{212} can be calculated using following Equation (3.15). **Figure 3.6** describes the coagulation process. Hamaker constant of some materials are listed in Table 3.2. $A_{212} = (A_{11}^{1/2} + A_{22}^{1/2} - A_{12}) / 2$ (3.15) $2 \times 10^{-21} \text{ J}$ $2 \times 10^{-21} \text{ J}$ $1 \times 10^{-21} \text{ J}$ + **Figure 3.6: The coagulation process of particles type-2 in medium type-1** **Table 3.1: Values of Debye parameter and inverse Debye parameter for several values of electrolyte concentrations and valences for aqueous solutions at 25 °C** **Symmetrical Electrolyte Molarity z+z- $\kappa \times (1m0-91) = z[M3.12/92 \times \kappa - 11(0m-10)1 = /2|z3| - 1.0M4 - 0.001 1:1 1.04 \times 10^8 2:2 2.08 \times 10^8 3:3 3.12 \times 10^8 0.01 1:1 3.29 \times 10^8 2:2 6.58 \times 10^8 3:3 9.87 \times 10^8 0.1 1:1 1.04 \times 10^9 2:2 2.08 \times 10^9 3:3 3.12 \times 10^9$** (Hiemenz & Rajagopalan, 1997) 9.61×10^{-9} 4.81×10^{-9} 3.20×10^{-9} 3.04×10^{-9} 1.52×10^{-9} 1.01×10^{-9} 9.61×10^{-10} 4.81×10^{-10} 3.20×10^{-10}

Asymmetrical Electrolyte $z+z- \kappa(m-1) = 2.32 \kappa^{-1}(m) = 4.30 \times 10^{-9} \times 109(\Sigma z_i^2 M_i)^{1/2} 10^{-1} (\Sigma z_i^2 M_i)^{-1/2} 1:2, 2:1$
 $1.80 \times 10^8 5.56 \times 10^{-9} 3:1, 1:3 2.54 \times 10^8 3.93 \times 10^{-9} 2:3, 3:2 4.02 \times 10^8 2.49 \times 10^{-9} 1:2, 2:1 5.68 \times 10^8 1.76 \times 10^{-9}$
 $1:3, 3:1 8.04 \times 10^8 1.24 \times 10^{-9} 2:3, 3:2 1.27 \times 10^9 7.87 \times 10^{-10} 1:2, 2:1 1.80 \times 10^9 5.56 \times 10^{-10} 1:3, 3:1 2.54 \times 10^9$
 $3.93 \times 10^{-10} 2:3, 3:2 4.02 \times 10^9 2.49 \times 10^{-10}$ Table 3.2: Hamaker constants and [Point of Zero Charge \(pzc\) of](#)
different materials Materials [Water](#) Al₂O₃ TiO₂ ZnO SiO₂ A($\times 10^{-20}$ J) pzc Sources 4.35 (Hiemenz & Rajagopalan, 1997) 15.4 8.1-9.7 (Hiemenz & Rajagopalan, 1997), (Butt et al., 2006) 15 2.9-6.4; (Lefevre & Jolivet, 2009), (Butt et al., 2006), 7.5 (Fernandez-Nieves et al., 1998)) 9.2 (Lefevre & Jolivet, 2009) 6.5 1.8-3.4 (Lefevre & Jolivet, 2009), (Butt et al., 2006) Sedimentation begins with the preparation and becomes dominating as soon as the particles agglomerated. Gravitational force becomes active and increases with the increment of growth in size of agglomerates. Under steady state condition using force equilibrium we can write the relation of buoyancy, resistance and gravitational forces acting on a single particle (Pabst & Gregorova, 2007) as Equation (3.16). The lift force (buoyancy force) acting on the particle and it can be determined using Equation (3.17). The resistance force acting on the particle exerted by the medium was calculated using Equation (3.18). Viscosity of basefluid is presented in Table A.1 in Appendix A. $F_b + F_r - F_g = 0$ (3.16) $F_b = nRa^3\rho L g$ (3.17) $F_r = 6\eta n R a v$ (3.18) The gravitational force acting on the particle was calculated using Equation (3.19), where we considered the aggregates' mass due to the difference between the solid and aggregate densities. Sedimentation velocity at steady state condition was calculated combining equations (3.16) to (3.19). Thus, the sedimentation velocity is presented as Equation (3.20). Equation (3.20) shows two forms of v; in first form, radius of gyration is replaced by equivalent radius for simplification and in the second form, mass of aggregates has been considered for gravitational force whereas radius of gyration is kept unchanged in buoyancy force. In this work, the first form considering equivalent radius is used. $F_g = 3 n R e^3 \rho_s g = m a g$ (3.19) $4 v = 2 R e^2 g (\rho_s - \rho_L) = m a g - 3 n R a \rho L g$ (3.20) $4 \times 3 \times 9 \times 6 \times \eta R a$ DLS system based equipment "Zetasizer zs" from Malvern, [as shown in Figure 3.7](#), was used to [measure size](#) distribution of [nanoparticles in](#) nanofluids as well as zeta potential of nanofluids at different time intervals after preparation. TEM (Leo-Libra 120), [as shown in Figure 3.8](#) was used to [measure](#) particle sizes and to observe particles' orientation in aggregates in the nanofluids. 3.4 Investigation of the Optical Properties of Nanofluids This section is divided in few sub-sections to describe methods of investigation of optical coefficients (absorption, scattering and extinction coefficients), refractive index and conversion of experimental data separately. Specific models and methods are presented broadly in their respective sub sections. Figure 3.7: Zetasizer zs Figure 3.8: TEM (Leo-Libra 120) 3.4.1 Optical Coefficients In this work, Rayleigh scattering (equations (3.21) to (3.29)) and Quasi Crystalline approach (Equation (3.30)) were selected for analytical analysis of [absorption, scattering and extinction coefficients](#). The approaches [are](#) discussed [in the](#) following subsections. 3.4.1.1 Rayleigh Approach (RA) To find out extinction coefficient as well as refractive index of a nanofluids, a theoretical model based on Rayleigh scattering approach has been adopted which was proposed by Taylor, et al. (2011). Assumptions of the model according to Taylor, et al. (2011) are as follows: a) Independent scattering b) All the particles are of same size and spherical c) Particle size is very small d) Lower volume concentration (<0.6 vol. %) e) Optical properties of nanoparticles are as same as their bulk materials f) Nanoparticles are of graphite or metal Non dimensional [particle size parameter \(a\)](#) is [defined as](#), $n_D a = \lambda$ (3.21) Working in independent scattering regime allows the scattering, absorption and extinction coefficients respectively for each nanoparticles to be formulated in the following manner. $Q_{scat} = a^4 m^2 + 2.8 m^2 - 1.23$ (3.22) $Q_{abs} = 4 \text{Im}[\epsilon] m^2 + 12 \text{Re}[\epsilon] m^2 + a^2 152 \text{Re}[\epsilon] m^2 + 12 \text{Re}[\epsilon] m^4 2m^2 72m^2 + 23 + 38 \text{Re}[\epsilon] m^2 + \text{Re}[\epsilon]$ (3.23) $Q_{ext} = Q_{abs} + Q_{scat}$ (3.24) Now, the extinction coefficient (σ_{ext}) of the particles can be represented as, $\sigma_{ext, particles} = 3\phi(Q_{abs} + Q_{scat}) / D$ (3.25) Comparing the equations (3.22) and (3.23) it is found that $Q_{scat} < \text{scattering and absorption coefficients can be}$ obtained [by](#) neglecting [the](#) absorption and scattering effect from Equation (3.25). $3\phi Q_{abs} / \sigma_{abs, particles} = 2 / D$ (3.26) $3\phi Q_{scat} / \sigma_{scat, particles} = 2 / D$ (3.27) During the formulation of the equations (3.22) to (3.27), working medium was considered fully transparent. However, base fluid or working medium has non negligible absorption [coefficient and it can be calculated](#) with [the following](#) proposed approach. $4n_{basefluid} \sigma_{basefluid} = \lambda$ (3.28) As a first order approximation (as proposed in the model), the [extinction coefficient of the](#) nanofluids [can be](#) found simply [by adding the extinction coefficients of](#) its base fluid [and](#) nanoparticles [as](#) shown in Equation (3.29). $\sigma_{total} = \sigma_{basefluid} + \sigma_{particles}$ (3.29) 3.4.1.2 Quasi Crystalline Approach (QCA) Multiple dependent scattering occurs in nanofluids and Quasi Crystalline Approximation is found more appropriate to calculate scattering coefficient (Prasher, 2005; Prasher & Phelan, 2005). According to the QCA, scattering coefficient can be obtained using Equation (3.30) (Prasher, 2005; Prasher & Phelan, 2005), where, $u = \frac{m^2 - 1}{m^2 + 2}$ and $\frac{m^2 + 2}{2n\rho a} = \lambda$. $\sigma_{scat} = (2u^2 a^4 / a) \phi (1 - \phi)^4 (1 + 2\phi)^2 (1 - \phi u) [1 + (3\phi u / (1 - \phi u))]$ (3.30) 3.4.2 Complex Refractive Index The [complex refractive index](#) of nanofluids [can be](#) obtained [using the](#) Maxwell-Garnett [effective medium calculation](#). Equation (3.31) shows the approach. $\epsilon_{eff} = \frac{\epsilon_p - \epsilon_f}{\epsilon_p + 2\epsilon_f} \phi + \epsilon_f$ (3.31) $\epsilon_{eff} = \frac{\epsilon_p - \epsilon_f}{\epsilon_p + 2\epsilon_f} \phi + \epsilon_f$ (3.31) $\epsilon_{eff} = \frac{\epsilon_p - \epsilon_f}{\epsilon_p + 2\epsilon_f} \phi + \epsilon_f$ (3.31) Once the [dielectric constant of the](#) effective medium (nanofluid) is obtained, [the](#) components [of the](#) refractive index [can be](#) calculated by using equations (3.32) and (3.33). All the above mentioned equations are obtained from Taylor et al. (2011). $n_{eff} = \sqrt{\epsilon_{eff}}$ (3.32) $k_{eff} = \sqrt{\epsilon''_{eff}}$ (3.33) 2 To calculate refractive index of Alumina and Titania nanofluids, dielectric constants of Alumina, Titania and water were obtained by converting the refractive index values (as shown in Table 3.3) of them according to the method described in Palik (1998). Refractive index of Alumina and Titania were taken from Palik (1998) and Refractive index database (2012) respectively and for [water from Hale & Querry \(1973\)](#). Though [the](#) scattering effect [for the](#) above computation has not been considered, it has significant effect especially when the particles are of larger size. The [fraction of incident light that is scattered](#) can be found using the Equation (3.34) (Taylor et al., 2011). $I_{scat} = \frac{n^4 ND^6 m^2 - 1}{(1 + \cos 2\theta)} I_0 \frac{8\lambda^4 r^2 m^2 + 2}{m^2 + 2} = (3.34)$ Table 3.3: Complex Refractive Index of Alumina, Titania and Water Wavelength [Real \(n\) and](#) Complex [\(k\) Parts of Refractive Index](#) λ (nm) Alumina Titania Water $n_k n_k n_k x$ 105 200 1. 91271 1.53584 1. 70000 1. 39600 0.01100 250 1.83370 1.36502 2.85000 1.36200 0.00335 300 1.81477 4.73162 3.28000 1.34900 0.00160 350 1.79712 4.47728 0.65000 1.34300 0.00065 400 1.78618 3.28609 1.33900 0.00019 450 1.77937 3.14054 1.33700 0.00010 500 1.77467 3.03000 1.33500 0.00010 550 1.77103 2.95438 1.33300 0.00020 600 1.76806 2.90000 1.33200 0.00109 650 1.76552 Null 2.86000 1.33100 0.00164 700 1.76336 2.83000 1.33100 0.00335 750 1.76147 2.81000 Null 1.33000 0.01560 800 1.75982 2.79000 1.32900 0.01250 850 1.75837 2.78000 1.32900 0.02930 900 1.75706 2.77000 1.32800 0.04860 950 1.75589 2.76063 1.32700 0.29300 1000 1.75487 2.75000 1.32700 0.28900 1050 1.75391 2.74739 1.32625 0.46400 1100 1.75306 2.74234 1.32550 0.63900 Source: Alumina from Palik (1998), Titania from Refractive index database (2012) and Water from Hale & Querry (1973). Experimentally, refractive index was measured for 589.3 nm wavelength at different temperatures (25-70°C) using Refractometer (RM-40 from Mettler Toledo) [as shown in Figure 3.9](#). Precision of [the](#) refractometer [is](#) 0.01%. Figure 3.9: Refractometer (RM-40 from Mettler Toledo) 3.4.3 Conversion of Transmittance to Extinction Coefficient Light extinction coefficient can be found from the [Lambert Beer law](#). According to [the law, the intensity of light](#) at a distance "r" can be written as the following relation (Mercatelli et al. The extinction (3.35) (3.36) al., 2011b). $\ln(I / I_0) = -\sigma_{ext} r$ or $\sigma_{ext} = \sigma_{abs} + \sigma_{scat} = \delta \sigma_{ext}$ coefficients. Mathematically it can be written as the following relation. n coefficient is a combination of the absorption and the scattering Experimental light of 200-1100 nm wavelengths (Spectrosil quartz, England) cuvettes were brought to ensure minimum visible spectroscopy (Lambda 35 was measured for I_y , extinction Transmittance Transmittance from Perkin prepared and measured in the spectroscopy at cons Elmer), as shown n coefficient was obtained by measuring transmittance from UV in Figure 3.10. ent using Equation (3.35). . Samples were tant temperature 25°C. Special quartz ss of

light within values were converted to extinction coefficient at the specified wavelength region. It should be noted that the material is claimed to have transmittance value over 90% for 200-2000 nm wavelength. Depth of the cuvettes were 10mm. Transmittance data were taken at one hour intervals after preparation of nanofluids to understand the effect of time on attenuation of light. Figure 3.10: UV visible spectroscopy (Lambda 35 from Perkin Elmer) 3.5 Development of a Mathematical Model There are two broad methods of reasoning such as deductive and inductive approaches. The deductive approach is used for the more general to the more specific works. In this method work is started from the general formula to get a specific result at the end for a particular subject and thus sometimes the method is referred to as "top-down" method. The inductive approach is just opposite in sense to the deductive approach; it is used for the most specific to the most general works. In this method observation is started from the most specific problem and is ended with the most general solution and thus sometimes it is referred to as "bottom-up" method. On the basis of the above information, mathematical modelling falls under the inductive reasoning approach. The [flow chart of the Inductive approach is shown in Figure 3.11](#). Different tensions were generated and became the part of the procedure to represent a real world phenomenon in mathematical format. Figure 3.12 represents the summary of the tensions in pictorial format. Data analysis is a process of examining, clearing, transforming and modelling to get or discover important information that has been buried in them. There are mainly three approaches for analysing data: Classical, Exploratory and Bayesian. All the [three approaches are similar in the sense that they start from general science or engineering problems and finally reach to science or engineering conclusion](#). However, difference among them lies in [the sequence and focus of intermediate steps](#). Figure 3.13 represents the sequence of intermediate steps in the approaches. Exploratory Data Analysis (EDA) was selected for our purpose because it properly matches with the inductive approach. Transmittance data were collected from the UV visible spectroscopy where data are presented after statistical analysis in the equipment. However, for more accuracy same data were taken few times. Later on, extinction coefficients at different time interval were plotted against wavelength, time and 46 Theory Tentative Hypothesis Pattern Determination Experimental Investigation Figure 3.11: Inductive approach of reasoning Figure 3.12: Summary of tensions from hypothetical experiment. (Source: Das, 2005) Classic Approach Exploratory Approach Bayesian Approach Problem Problem Problem Data Data Data Model Analysis Model Analysis Model Prior Distribution Conclusion Conclusion Analysis Conclusion Figure 3.13: Sequence of steps in different data analysis approaches concentration. Aggregation parameters were also used for estimating extinction coefficients at different stages of aggregation. After that, all the information were carefully investigated in Microsoft Excel. To initiate the development of a new model, the theoretical values based on existing models with experimentally obtained values were compared. Table 2.2 in Chapter 2 describes the advantages and limitations of the present models available. Keeping in mind about the limitations of previous models and differences in experimental and estimated values, relations among different optical and aggregation parameters were studied carefully for the existence of any possible relation. Thus, aforementioned information points towards combining aggregation phenomena and optical property calculation approach together to get cumulative realistic calculation approach of optical properties. Now, on the basis of above discussion, optical properties of nanofluids can be defined as [a function of optical properties of materials, particles' volume concentration and particle concentration in aggregates](#). The definition is mathematically presented in Equation (3.37), $P(t) = f(\phi_{int}, \phi_a, \phi_p, P_0, P_t, P_a)$ (3.37) where $P(t)$ is the optical property of nanofluid at time t , P_0 is the optical property of nanofluid at time $t=0$ i.e. when particles are all separated, P_t and P_a are the optical properties at time t and for the aggregates respectively. 3.6 Error Analysis Deviation from the experimental value is considered as an error in the estimated values and it is presented in percentage. Mathematically it can be written as follows: (3.38) 3.7 Summary of Chapter 3 This chapter broadly describes the methods which were applied during the study. The two-step method of preparation, electrostatic stability analysis technique along with time variant aggregation phenomena, approaches for determining and comparing optical properties such as, Rayleigh, Quasi Crystalline, Maxwell-Garnett and Lambert-Beer approaches, and data analysis procedure based on inductive approach and EDA system are broadly discussed and they are interlinked to present a complete method of the work. Results of this work based on the above mentioned method [are presented in the Chapter 4 and discussed afterward in the Chapter 5. CHAPTER 4: RESULTS 4.1 Introduction This chapter focuses on the presentation of results of the experiments and analysis that were obtained based on the methodology described in Chapter 3. The data obtained throughout the investigation are interpreted and presented in tabular and pictorial format. This chapter is divided in three sections and several subsections based on the objectives of the study. 4.2 Results of Stability of Nanofluids Results of stability analysis of the nanofluids have been presented in this section. The following sub-sections present the stability analysis report of Alumina and Titania nanofluids based on Section 3.3. 4.2.1 Stability of the Alumina Nanofluids: Results showed that the stability ratio for the Alumina nanofluids were at least 100 times higher than the threshold limit \(105\) for 0.05 %v/v and 0.08 %v/v, whereas approximately 1000 times higher for 0.03 %v/v. Time constants of all the considered nanofluids were large enough to conduct optical tests as they were found with time constant higher than 2 hours. Time constants, stability ratio, zeta potential and fractal dimensions are shown in Table 4.1. The nanofluid of 0.05 %v/v was considered for TEM imaging to discover growth of nanoparticles. Table 4.1: Aggregation parameters and Stability ratio of the Alumina nanofluids Dia. of Conc. pH Zeta Potential Fractal Time const., \$\tau_p\$ Stability ratio NPs \(nm\) \(%v/v\) \(mV\) Dimension \(hr\) \(W\) \$\times 10^{-6}\$ 0.03 9 54.47 1.5 41 237 13nm 0.05 9 52.25 1.7 5.16 49.7 0.08 9 51.84 1.9 2.44 37.6 Table 4.2 presents sedimentation rate and size of aggregates in the Alumina nanofluids. Sedimentation rate was found increasing with time and concentration. Figure 4.1 shows experimentally \(from the equipment Zetasizer that uses DLS technique\) obtained size distribution graph of the aggregates in the nanofluid at different times after preparation. Peaks of the distributions are at almost identical horizontal position; however, their intensity increases vertically conveying the information that the population of aggregates was increased. The intensity was increased by 0.73% within eight hours after preparation. Aggregation process \(step by step\) is clearly represented in TEM images in \[Figure 4.2 and Figure 4.3\]\(#\). Observing the TEM images we find that two primary clusters of nanoparticles facing each other form a secondary cluster. Sizes of the aggregates from both TEM image and DLS approach were found in good agreement as they fall in same range. Table 4.2: Sedimentation rate and size of aggregates in the Alumina nanofluid Sedimentation rate \(m/sec\) \$\times 10^{10}\$ da from DLS \(nm\) Time, t \(hr\) Concentrations \(%v/v\) Concentrations \(%v/v\) da from TEM \(nm\) 0.03 0.05 0.08 0.03 0.05 0.08 1 3.16 3.49 3.90 127.9 138.0 127.1 4 3.30 4.55 5.93 132.4 137.5 124.9 120-140 6 3.40 5.19 7.10 125.9 135.4 126.0 Intensity \(%\) 16 14 12 10 8 At 2.5 hour At 6.5 hour At 8.4 hour 6 4 2 0 10 100 Diameter of Nanoparticles \(nm\) Figure 4.1: DLS size distribution of the clusters in 0.05%v/v Alumina nanofluid A B C D Figure 4. 2: TEM images at 1st, 2nd and 4th hours after preparation of Alumina nanofluid. A\) Initialization of primary cluster formation at 1st hour, B\) Inter distance among the primary clusters at 2nd hour and C, D\) Primary clusters at 4th hour A B C Figure 4.3: TEM images on 10th day after preparation of Alumina nanofluid. A\) Initial stage of secondary cluster, B\) Development of secondary cluster and C\) Relative position of secondary clusters 4.2.2 Stability of the Titania Nanofluids: In the case of Titania nanofluids, obtained stability ratio were found sharply decreasing with increase in concentration of nanoparticles. The most favourable stability was found for 0.03 %v/v, which was 1000 times higher than the threshold limit \(105\) followed by 0.05 %v/v where it was 100 times higher. For 0.08 %v/v, the ratio was estimated 10 times higher than the threshold limit. Time constants for 0.03 %v/v and 0.05 %v/v were found favourable to conduct optical tests as the time constants were more than 2 hours. Zeta potential, fractal dimensions, time constants and stability ratio for the Titania nanofluids \[are shown in Table 4.3. The nanofluid of 0.05 %v/v was considered for TEM imaging to\]\(#\)](#)

discover growth of nanoparticles. Table 4.4 shows the sedimentation rate of aggregates in the nanofluids at different times and concentrations along with observed size distributions. Sedimentation rate is found increasing with time and concentration. Table 4.3: Aggregation parameters and stability ratio of the Titania nanofluid at 25°C Dia. of NPs Conc. pH Zeta Potential Fractal Time const., Stability ratio (nm) (%v/v) (mV) Dimension tp (hr) (W)×10-6 0.03 4 45.33 1.32 65.2 104 20nm 0.05 4 43.9 1.58 11.5 30.5 0.08 4 42.14 1.80 1.7 7.19 Table 4.4: Sedimentation rate and size of aggregates in the Titania nanofluids at 25°C Sedimentation rate (m/sec) ×1010 da from DLS (nm) Time, t (hr) Concentrations (%v/v) Concentrations (%v/v) da from TEM (nm) 0. 03 0.05 0. 08 0.03 0. 05 0. 08 1 8.07 8.44 10.9 159.7 158.9 162 4 8.31 9.74 17.9 163.6 175.4 165.9 130-170 6 8.47 10.6 21.9 158.0 160.0 167.2 16 14 After 1 Hour 12 After 4 Hours After 6 Hours Intensity (%) 10 8 6 4 2 0 10 100 1000 Size (nm) Figure 4.4: DLS size distribution of the clusters in 0.05 %v/v Titania nanofluid Figure 4.5: TEM images of Titania nanofluid. Figure 4.4 shows size distribution graph of the aggregates in the Titania nanofluid at different times after preparation. Peaks of the distributions are at almost identical horizontal position for last two sampling whereas the peak for the first hour is slightly left shifted. Although at the first hour intensity was higher than the others peak position was at smaller size. Size of the aggregates started increasing with time and as a result their population increased as well. Figure 4.5 shows the TEM images of the TiO2 nanoparticles inside the nanofluid. Therefore, it can be said that population of randomly moving single particles decreases with time and form aggregates in both Alumina and Titania nanofluids. The population of aggregates increases with time till all the single particles are transformed into aggregates. 4.3 Results of Investigation on Optical Properties of Nanofluids This section contains results of both analytical and experimental investigation of optical properties (e.g. extinction, absorption and scattering coefficients, transmittance and refractive index) of the Alumina and Titania nanofluids. The section is divided into two sub-sections for describing the results of Alumina and Titania nanofluids separately. 4.3.1 Optical Properties of Alumina Nanofluids: Scattering coefficients of the Alumina nanofluids, which were obtained from RA and QCA, are presented in Table 4.5 to be able to compare the values. Differences between the estimated values with the above mentioned analytical approaches are very small and thus can be neglected. For the negligible difference between the values convey the message about single independent scattering of light by the Alumina particles in the base medium. Therefore, selection of Rayleigh Approach for analytical work is justified. Figure 4.6 presents the scattering and absorption coefficients of Alumina nanoparticles of various sizes based on the Rayleigh approach. It was observed from the analysis that 55 size of the nanoparticles had significant effect on scattering coefficient. Scattering coefficient of Alumina nanofluids increases both with increase in size and concentration. Although, the values of scattering coefficients, for smaller than 10 nm diameter, were found negligible (values times 10-3-10-5) prominent increase in the value was observed for 10 nm or above. It becomes the most important factor for the nanoparticles of above 20 nm diameter. On the other hand, absorption coefficients for all the sizes were found almost equal for same concentrations. Increase in it was observed in the region of 200-400 nm and above 550 nm with increase in concentrations. For all the three considered concentrations, absorption coefficients were found approximately zero within 400-550 nm wavelength region where peak of the solar spectrum is laying. Overall absorption coefficient of Alumina nanoparticles were negligibly small. Figure 4.7 demonstrates the analytical result of extinction coefficients of the Alumina nanofluids of 0.03, 0.05 and 0.08 %v/v concentrations. Maximum value of 0.64 cm-1 was obtained for 0.08 %v/v at 200 nm whereas others remain below 0.6 cm-1 at that wavelength. The values fall dramatically to 0.29 cm-1, 0.18 cm-1 and 0.11 cm-1 at 250 nm; very slow variation was observed after 250 nm wavelength. However, it became identical for all the concentrations after 550 nm wavelength. Experiment was conducted to measure extinction coefficients of the samples and the result for the first and sixth hours after preparation is presented in the Figure 4.8. The complete experimental transmittance values at different time after preparation are given in Table A.2 in Appendix A. We found similar trend of curves of extinction coefficient as estimated in analytical analysis. However, a huge difference in the experimental and analytical values of extinction coefficient was perceived. Experimental data were found more than 10 times higher in UV region whereas in the visible and IR region the values lowered down from 10 to 1 times. Extinction coefficient reaches 6 cm-1 at 200 nm wavelength and dramatically fall down to 0.9 cm-1 at 400 nm for 0.03 %v/v concentration. At the first hour, in visible region (400-700 nm), extinction coefficient of water (basefluid) was enhanced by averagely 5.5, 9.5 and 18.7 times for 0.03, 0.05 and 0.08 %v/v. Table 4.5: Comparison of scattering coefficients of the Alumina nanofluids: estimated using RA and QCA for 13 nm diameter nanoparticles Conc. (%v/v) λ(nm) 0.03 σscat(cm⁻¹)×102from RA 0.05 0.08 0.03 σscat(cm⁻¹)×102from QCA 0.05 0.08 0.03 Differences×102 0.05 0.08 200 8.217 13.695 21.912 8.197 13.639 21.770 0.020 0.055 0.142 250 2.987 4.978 7.964 2.979 4.957 7.913 0.007 0.020 0.051 300 1.432 2.387 3.819 1.429 2.378 3.795 0.003 0.010 0.025 350 0.744 1.241 1.985 0.743 1.236 1.972 0.002 0.005 0.013 400 0.427 0.711 1.138 0.426 0.708 1.130 0.001 0.003 0.007 450 0.007 0.012 0.019 0.007 0.012 0.019 0.000 0.000 0.000 500 0.170 0.284 0.454 0.170 0.283 0.451 0.000 0.001 0.003 550 0.116 0.193 0.309 0.116 0.192 0.307 0.000 0.001 0.002 600 0.081 0.135 0.217 0.081 0.135 0.215 0.000 0.001 0.001 650 0.059 0.098 0.157 0.059 0.097 0.156 0.000 0.000 0.001 700 0.043 0.072 0.115 0.043 0.072 0.115 0.000 0.000 0.000 750 0.033 0.055 0.087 0.033 0.054 0.087 0.000 0.000 0.001 800 0.025 0.042 0.067 0.025 0.042 0.067 0.000 0.000 0.000 850 0.020 0.033 0.053 0.020 0.033 0.052 0.000 0.000 0.000 900 0.016 0.026 0.042 0.016 0.026 0.042 0.000 0.000 0.000 950 0.013 0.021 0.034 0.013 0.021 0.033 0.000 0.000 0.000 1000 0.010 0.017 0.027 0.010 0.017 0.027 0.000 0.000 0.000 1050 0.008 0.014 0.022 0.008 0.014 0.022 0.000 0.000 0.000 1100 0.007 0.012 0.019 0.007 0.012 0.019 0.000 0.000 0.000 A1 1.0E-04 Scattering Coefficients (1/cm) 8 0.E-05 6 0.E-05 4 0.E-05 2.0E-05 For 0.03 %v/v For 0.05 %v/v For 0.08 %v/v A2 5.0E-05 4.0E-05 3.0E-05 2.0E-05 1.0E-05 Absorption Coefficients (1/cm) 0.0E+00 200 300 500 Wavelength (nm) 400 600 700 0.0E+00 200 300 B 1 For 0.03%v/v B 2 1.5E-02 5.0E-05 Scattering Coefficients (1/cm) 1.2E-02 For 0.05%v/v For 0.08%v/v Absorption Coefficients (1/cm) 4.0E-05 9.0E-03 3 0.E-05 6.0E-03 2 0.E-05 3.0E-03 1 0.E-05 0.0E+00 200 300 400 500 600 700 200 300 0.0E+00 Wavelength (nm) Figure 4.6, Continued 58 400 500 Wavelength (nm) 400 500 Wavelength (nm) For 0.03 %v/v For 0.05 %v/v For 0.08 %v/v 600 700 For 0.03 %v/v For 0.05%v/v For 0.08 %v/v C 1 0.10 For 0.03%v/v Scattering Coefficients (1/cm) 0.08 For 0.05%v/v For 0.08 %v/v 0.06 0.04 0.02 0.00 200 300 400 500 600 700 Wavelength (nm) D 1 0.24 For 0.03%v/v Scattering Coefficients (1/cm) 0.20 For 0.05%v/v For 0.08%v/v 0.16 0.12 0.08 0.04 0.00 200 300 400 500 600 700 Wavelength (nm) Figure 4.6, Continued C 2 5.0E-05 Absorption Coefficients (1/cm) 4.0E-05 3.0E-05 2.0E-05 1.0E-05 0.0E+00 For 0.03%v/v For 0.05%v/v For 0.08%v/v 200 300 400 500 Wavelength (nm) 600 700 200 300 Wavelength (nm) 400 500 600 700 D 2 5.0E-05 Absorption Coefficients (1/cm) 0.0E+00 4.0E-05 3.0E-05 2.0E-05 1.0E-05 For 0.03 %v/v For 0.05 %v/v For 0.08%v/v 59 E1 1.00 Scattering Coefficients (1/cm) 0.80 0.60 0.40 0.20 0.00 For 0.03 %v/v For 0.05 %v/v For 0.08 %v/v E 2 5.0E-05 Absorption Coefficients (1/cm) 4.0E-05 3.0E-05 2.0E-05 1.0E-05 0.0E+00 For 0.03%v/v For 0.05%v/v For 0.08%v/v 200 300 400 500 600 700 200 300 400 500 600 700 Wavelength (nm) Figure 4.6: Scattering (left) and absorption (right) coefficients of Alumina nanoparticles as estimated using the Rayleigh approach at 0.03, 0.05 and 0.08 %v/v concentrations and for A) 1 nm, B) 5 nm, C) 10 nm, D) 13 nm and E) 20 nm diameters. 60 0.80 For 0.03 %v/v For 0.05 %v/v For 0.08 %v/v Extinction Coefficient (1/cm) 0.60 0.40 0.20 0.00 200 300 400 500 600 700 800 900 1000 1100 Wavelength (nm) Figure 4.7: Extinction coefficient of the Alumina (13 nm) nanofluids of different concentration from Rayleigh Approach. Figure 4.9 illustrates the changes in extinction behaviour of the Alumina nanofluids within first six hours of preparation. Less than 5% reduction in the extinction coefficient was noticed in UV region whereas sharp increase in the reduction was observed from 400-900 nm. Sudden fall in reduction occurred after 900 nm wavelength. This information on the reduction pin point the visible and near IR region as

critical zone. Although extinction coefficient within this region was found below 3 cm⁻¹, the reduction was found varying (visible to IR) from 6-44% for 0.05 %v/v concentration followed by 5-35% for 0.03%. The value for 0.08 %v/v Alumina nanofluid was found the lowest in variation; for this sample, within wavelength 400-900 nm, the reduction value was 50- 20% lower than the corresponding value of 0.05 %v/v Alumina nanofluids. Table 4.6 presents the values of analytically obtained complex refractive index of the Alumina nanofluids from the effective medium approach. Refractive indexes of the nanofluids were experimentally measured for different temperatures at 589.3 nm wavelength and the result has been shown in Figure 4.10. It is observed from the Figure 4.10 that refractive index was decreasing with an increase in temperature. 0.067% increase in the value of n was noted for 0.05 %v/v Alumina nanofluids compared to distilled water. Although n was found increasing with an increase in concentration from 0.03 to 0.05 %v/v, values of n for 0.08 %v/v concentration lied between other two concentrations. Figure 4.11 presents the difference between analytical and experimental values of the refractive index (n). Experimental results of n showed higher values than the Maxwell-Garnett estimation at 589.3 nm wavelength. 8 At first hour for 0.03%v/v 7 At first hour for 0.05%v/v Extinction Coefficients (1/cm) At first hour for 0.08%v/v 6 Basefluid 5 At sixth hour for 0.03%v/v At sixth hour for 0.05%v/v 4 At sixth hour for 0.08%v/v 3 2 1 0 200 300 400 500 600 700 [800 900 1000 Wavelength \(nm\) Figure 4.8](#): Experimental extinction coefficient of the Alumina nanofluids at different wavelength and concentrations 50 Reduction for 0.03% 45 Reduction for 0.05% Reduction in Ext. Coefficients (%) 40 Reduction for 0.08% 35 30 25 20 15 10 5 0 200 300 400 500 [600 700 800 900 1000 Wavelength \(nm\) Figure 4.9](#): Reduction in extinction coefficients within first six hours after preparation of the Alumina nanofluids Table 4.6: Complex Refractive Index of water based Alumina nanofluids at 25°C Concn. (%v/v) λ (nm) 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950 1000 1050 1100 1150 1200 1250 1300 1350 1400 1450 1500 0.03 1.396142 1.362131 1.349129 1.343126 1.339124 1.337123 1.335122 1.333122 1.332121 1.331121 1.331120 1.330120 1.329120 1.329120 1.328120 1.327120 1.327119 1.326369 1.325619 1.324869 1.324119 1.323369 1.322619 1.321869 1.321119 1.320119 1.319119 [N.O. 05 1. 396237 1. 362218 1. 349215 1. 343210 1. 339207 1. 337205 1. 335204 1. 333203 1. 332202 1. 331201 1.330200 1.329200 1.329199 1.328199 1.327199 1.327199 1.326449 1.325699 1.324949 1.324199 1.323449 1.322699 1.321949 1.321199 1.320199 1.319199 0.08 1.396379 1.362348 1.349344 1.343336 1.339331 1.337328 1.335326 1.333325 1.332324 1.331323 1.331321 1.330321 1.329320 1.329319 1.328319 1.327319 1.327318 1.326568 1.325818 1.325068 1.324318 1.323568 1.322818 1.322068 1.321318 1.320318 1.319318 0.03 0.001110 0.000333 0.000149 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000 0.000105 0.000149 0.000333 0.001538 0.001242 0.002933 0.004856 0.029307 0.028893 0.046398 0.063885 0.081392 0.098881 0.419092 0.739251 1.059433 1.379676 1.248405 1.117239 \$k \times 10^4\$ 0.05 0.001110 0.000333 0.000149 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000 0.000105 0.000149 0.000333 0.001538 0.001242 0.002933 0.004855 0.029302 0.028888 0.046390 0.063874 0.081378 0.098864 0.419023 0.739130 1.059259 1.379450 1.248201 1.117056 0.08 0.001110 0.000333 0.000149 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000 0.000105 0.000149 0.000333 0.001541 0.001238 0.002931 0.004854 0.029295 0.028881 0.046379 0.063858 0.081358 0.098840 0.418920 0.738948 1.058999 1.379111 1.247894 1.116781 1.334 1.333 \[1.332 Refractive Index 1.331 1.330 1.329 1.328 1.327 1.326\]\(#\) 0.03 %v/v 0.05 %v/v 0.08%v/v Water 1.325 20 30 40 \[50 60 70 Figure 4.10\]\(#\): Refractive index of the Alumina nanofluids at different temperatures and Temperature\(°C\) concentrations 1.3334 Experimental Maxwell-Garnett's Effective Medium Approach 1.3332 Refractive Index \(n\) \[1. 333 1. 3328 1. 3326 1. 3324 0. 03% 0. 04% 0. 05% 0. 06% 0. 07% 0. 08% 0.09% Concentration \\(by volume\\) Figure 4.11\]\(#\): Comparison between experimental and analytical values of refractive index of the Alumina nanofluids at 589.3 nm wavelength and 25°C temperature 4.3.2 Optical Properties of Titania Nanofluids: Scattering coefficients of the Titania nanofluids which were obtained from RA and QCA are presented in Table 4.7 to be able to compare the values. Differences between the estimated values with the above mentioned approaches are very high for light of 200-350 nm wavelength region. RA estimates 1.4 to 200 times higher value than the QCA in the above mentioned region for 0.03 to 0.08 %v/v concentrations. However, for the rest of the region, the differences are negligible and sometimes zero. To be able to compare and relate behaviour of both of the nanofluids, the RA was selected for further calculations here as well. Table 4.7: Comparison of scattering coefficients of Titania nanofluids estimated from RA and QCA Concn. \(%v/v\) λ\(nm\) 0.03 σ_{scat}\(cm⁻¹\) from RA 0.05 0.08 200 5.060 8.433 13.493 250 4.513 7.522 12.035 300 1.022 1.704 2.726 350 0.382 0.636 1.018 400 0.143 0.239 0.382 450 0.082 0.137 0.220 500 0.050 0.084 0.134 550 0.033 0.055 0.087 600 0.022 0.037 0.059 650 0.016 0.026 0.042 700 0.011 0.019 0.030 750 0.008 0.014 0.023 800 0.006 0.011 0.017 850 0.005 0.008 0.013 900 0.004 0.007 0.011 950 0.003 0.005 0.008 1000 0.003 0.004 0.007 1050 0.002 0.004 0.006 1100 0.002 0.003 0.005 σ_{scat}\(cm⁻¹\) from QCA 0. 03 0. 05 0. 08 0. 03 0. 05 0. 08 0. 025 0. 042 0. 067 5.035 8.391 13.426 0.000 0.000 0.000 4.513 7.522 12.035 0.719 1.197 1.910 0.303 0.507 0.816 0.370 0.615 0.982 0.012 0.021 0.036 0.143 0.238 0.379 0 0.001 0.003 0.082 0.137 0.218 0 0 0.002 0.050 0.084 0.134 0 0 0 0.033 0.054 0.087 0 0.001 0 0.022 0.037 0.059 0 0 0 0.016 0.026 0.041 0 0 0.001 0.011 0.019 0.030 0 0 0 0.008 0.014 0.022 0 0 0.001 0.006 0.011 0.017 0 0 0 0.005 0.008 0.013 0 0 0 0.004 0.007 0.011 0 0 0 0.003 0.005 0.008 0 0 0 0.003 0.004 0.007 0 0 0 0.002 0.003 0.006 0 0 0 0.002 0.003 0.005 0 0 0 Differences \(RA-QCA\) Figure 4.12 shows the analytical results of scattering and absorption coefficients of Alumina nanoparticles that obtained from the Rayleigh Approach. This analysis had been done for the sizes of 1, 5, 10 and 20 nm diameters of the particles. Although the absorption coefficients were observed increasing with concentration no effect of nanoparticle size was observed on them. However, scattering coefficients were found increasing with both concentration and size of nanoparticles. Though the scattering coefficients are negligible compared to absorption coefficients for 1 and 5 nm sizes it became countable for the size higher than 10 nm in diameter. Nevertheless, scattering coefficients are very small in comparison with absorption coefficients for all the considered sizes and concentrations. Absorption coefficients reach to hundreds and become negligibly small within UV region. Figure 4.13 demonstrates the analytical result of extinction coefficients of the Titania nanofluids of 0.03, 0.05 and 0.08 %v/v concentrations. Maximum values, 656 \[cm-1\]\(#\), 282 \[cm-1 and\]\(#\) 189 \[cm-1\]\(#\) were obtained at 200 nm for the above mentioned concentrations respectively. The values fall dramatically to 0.47 \[cm-1\]\(#\), 0.329 \[cm-1 and\]\(#\) 0.233 \[cm-1\]\(#\) at 400nm; very slow variation was observed in the values till 900 nm wavelength. However, they became identical after 900 nm wavelength. Figure 4.14 presents the extinction coefficients of Titania nanofluids of different concentrations at the first and sixth hour after preparation. Although TiO₂ is considered opaque for UV radiation, we have found them becoming opaque for visible region at higher concentration as well. Extinction coefficient reaches 9.1 cm⁻¹ at 400 nm wavelength for 0.03 %v/v concentration. At the first hour, in visible region \(400-700 nm\), extinction coefficient of water \(basefluid\) was enhanced by averagely 89.5, 107 and 116 times for 0.03, 0.05 and 0.08 %v/v respectively. A1 2.0E-03 Scattering Coefficients \(1/cm\) \[1.6E-03 1.2E-03 8.0E-04 4.0E-04 0.0E+00\]\(#\) 200 300 B1 0.25 Scattering Coefficients \(1/cm\) \[0.20 0.15 0.10 0.05 0.00\]\(#\) 200 300 Figure 4.12, Continued \[400 500 Wavelength \\(nm\\) 400 500 Wavelength \\(nm\\)\]\(#\) For 0.03% v/v For 0.05% v/v For 0.08% v/v 600 For 0.03% v/v For 0.05% v/v For 0.08% v/v 600 700 700 A2 700 Absorption Coefficients \(1/cm\) \[600 500 400 300 200 100 0\]\(#\) 200 300 B2 700 Absorption Coefficients \(1/cm\) \[600 500 400 300 200 100 0\]\(#\) 200 \[300 400 500 Wavelength \\(nm\\) 400 500 Wavelength \\(nm\\)\]\(#\) For 0.03% v/v For 0.05% v/v For 0.08% v/v 600 700 For 0.03% v/v For 0.05% v/v For 0.08% v/v 600 700 67 C1 2.00 For 0.03% v/v Scattering Coefficients \(1/cm\) 1.60 For 0.05% v/v For 0.08% v/v \[1.20 0.80 0.40 0.00\]\(#\) 200 300 400 500 600 700 Wavelength \(nm\) D1 15.0 For 0.03% v/v For 0.05% v/v Scattering Coefficients \(1/cm\) 12.0 For 0.08% v/v \[9.0 6.0 3.0 0.0\]\(#\) 200 \[300 400 500\]\(#\) 600 700 \[Wavelength \\(nm\\) C2 700 Absorption Coefficients \\(1/cm\\) 600 500 400 300 200 100 0\]\(#\) For 0.03% v/v For 0.05% v/v For 0.08% v/v 200 D2 700 \[0\]\(#\) Absorption Coefficients \(1/cm\) 600 \[0 500 0 400 0 300 0 200 0 100 0 0\]\(#\) 200 \[300 400 500\]\(#\)](#)

[Wavelength \(nm\) 300 400 500 Wavelength \(nm\) 600](#) For [0.03% v/v](#) For [0.05% v/v](#) For [0.08% v/v](#) 600 700 700 Figure 4.12: Scattering (left) and absorption (right) coefficients of Titania nanoparticles as estimated using the Rayleigh approach at 0.03, 0.05 and 0.08 %v/v concentrations and for [A\) 1 nm, B\) 5 nm, C\) 10 nm, and D\) 20 nm](#) diameters. 68 Again, reduction in extinction coefficient within first six hour after preparation for 0.05 %v/v concentration was comparatively found more prompt than 0.03 and 0.08 %v/v in both visible and IR region as shown in the Figure 4.15. In visible region, 2.8% reduction in extinction coefficient was observed for 0.05 %v/v whereas the reduction for 0.08 and 0.03 %v/v were 2.5 and 1% respectively. At near IR region the extinction coefficient reduced by more than 10% for 0.05 %v/v whereas the value remained under 10% for other two concentrations. Extinction Coefficients of NF (1/cm) 1,000.00 100.00 10.00 1.00 0.10 For [0.03%v/v](#) For [0.05%v/v](#) For [0.08%v/v](#) 0.01 200 300 400 500 600 700 800 [900 1000 1100 Wavelength \(nm\) Figure 4. 13: Extinction coefficient of the](#) Titania (20 nm) nanofluids of different concentration from Rayleigh Approach 12 11 10 Extinction Coefficients (1 /cm) [9 8 7 6 5 4 3 2 1 0](#) 400 500 600 700 800 Wavelength (nm) At first hour for 0.03%v/v At first hour for 0.05%v/v At first hour for 0.08%v/v Basefluid At sixth hour for 0.03%v/v At sixth hour for 0.05%v/v At sixth hour for 0.08%v/v 900 1000 1100 Figure 4.1 4: Experimental extinction coefficient of the Titania nanofluids at different wavelength and concentrations 20 Reduction for 0.03%v/v Reduction for 0.05%v/v Reduction in Ext. Coefficients of NF (%) Reduction for 0.08%v/v 15 10 5 0 400 500 600 700 800 [900 1000 1100 Wavelength \(nm\) Figure 4. 15: Reduction in extinction coefficients within first six hours after preparation of the Titania nanofluids](#) Table 4.8: Complex Refractive Index of water based Titania nanofluids at 25°C containing 20 nm sized (diameter) nanoparticles from Effective Medium Approach Conct. (%v/v) λ (nm) 0.03 n 0.05 0.08 0.03 $k \times 10^4$ 0.05 0.08 200 1.396306 1.396510 1.396816 4.96261 8.27066 13.2332 250 1.362742 1.363237 1.363979 3.96823 6.61492 10.58711 300 1.349565 1.349941 1.350506 0.86001 1.43347 2.29398 350 1.343472 1.343786 1.344259 0.33238 0.55398 0.88648 400 1.339377 1.339629 1.340006 0.00000 0.00000 0.00000 450 1.337362 1.337603 1.337964 0.00000 0.00000 0.00000 500 1.335349 1.335581 1.335930 0.00000 0.00000 0.00000 550 1.333339 1.333566 1.333905 0.00000 0.00000 0.00000 600 1.332333 1.332554 1.332887 0.00011 0.00011 0.00011 650 1.331327 1.331546 1.331873 0.00015 0.00015 0.00015 700 1.331323 1.331540 1.331862 0.00033 0.00033 0.00033 750 1.330321 1.330534 1.330855 0.00154 0.00154 0.00154 800 1.329318 1.329530 1.329848 0.00124 0.00124 0.00124 850 1.329317 1.329528 1.329844 0.00294 0.00294 0.00294 900 1.328315 1.328525 1.328841 0.00486 0.00486 0.00486 950 1.327314 1.327523 1.327837 0.02931 0.02931 0.02931 1000 1.327312 1.327521 1.327833 0.02890 0.02890 0.02890 1050 1.326562 1.326770 1.327083 0.04641 0.04640 0.04640 1100 1.325811 1.326019 1.326331 0.06390 0.06389 0.06389 1150 1.325060 1.325268 1.325579 0.08141 0.08140 0.08140 1200 1.324310 1.324517 1.324827 0.09890 0.09890 0.09889 1250 1.323560 1.323766 1.324076 0.41917 0.41915 0.41913 1300 1.322809 1.323016 1.323325 0.73939 0.73936 0.73931 1350 1.322059 1.322265 1.322574 1.05963 1.05959 1.05952 1400 1.321309 1.321514 1.321823 1.37993 1.37988 1.37979 1450 1.320308 1.320513 1.320822 1.24864 1.24859 1.24851 1500 1.319307 1.319512 1.319819 1.11744 1.11740 1.11733 1.337 0.03%v /v 0.05%v/v 1. 335 0.08 %v/v Wa ter [Refractive Index 1. 333 1. 331 1.329 1. 327 1. 325 20 25 30 35 40 45 50 55 60 65 70 Temperature\(°C\) Figure 4.16: Experimental refractive index of the Titania nanofluids at different temperatures and concentrations 1.3355 Experimental Maxwell-Garnett's Effective Medium Approach 1.3350 Refractive Index \(\$n\$ \) 1. 3345 1. 3340 1. 3335 1. 3330 1. 3325 0. 03% 0. 04% 0. 05% 0. 06% 0. 07% 0.08% 0.09% Concentration \(by volume\) Figure 4.17: Comparison between experimental and analytical values of refractive index of the Titania nanofluids at 589.3 nm wavelength and 25°C temperature. Table 4.8 presents the values of analytically obtained complex refractive index of the Titania nanofluids from the effective medium approach. Result of refractive index measurement for different temperatures at 589.3 nm wavelength has been \[shown in Figure 4.16. It\]\(#\) is observed from the Figure 4.16 that refractive index \(\$n\$ \) was decreasing with an increase in temperature. 0.2% increase in the values of \$n\$ was noted for 0.05 %v/v Titania nanofluids compared to distilled water. Although \$n\$ was found increasing with an increase \[in concentration from 0. 03 to 0. 05 %v/v\]\(#\), values of \$n\$ for 0.08 %v/v concentration lied between other two concentrations. Figure 4.17 presents the difference between analytical and experimental values of \$n\$. Experimental values of refractive index were found higher than the estimate of Maxwell-Garnett approximation. 4.4 Mathematical Model Results from the previous sections were analysed and organized to obtain a generalized time dependent formula to estimate extinction coefficient of the nanofluids. The formula is presented as Equation \(4.1\), where \[A and B are\]\(#\) the \[constants\]\(#\). Values of the constants are presented \[in Table 4.9\]\(#\). Proposed model includes the methods which are described in sections 3.3-3.5 and reaches at its final formula at Equation \(4.1\). The experimental and proposed model's estimated values of extinction coefficients of the nanofluids are \[presented in Figure 4. 18 to Figure 4. 22\]\(#\). It should \[be\]\(#\) noted \[that the\]\(#\) model properly explains Alumina nanofluids' behaviour till 200 nm wavelength but it can explain the behaviour of Titania nanofluid till 650 nm wavelength. \$\sigma_{ext} = A.e^{\(1 - \phi_{int}\) - 2 \ln \phi} + B \phi^{\alpha} \sigma_{ext, \lambda}\$ \(4.1\) Table 4.9: Constants A and B for the Alumina and Titania nanofluids Alumina Nanofluids Titania Nanofluids Time, t \(hr\) A B A B Concentrations, \$\phi\$ \(%v /v\) \[0.03 0.05 0.08 0. 03 0. 05 0. 08 0. 03 0. 05 0. 03 0. 05 1 0 8 0.9 1.0 5.6 3.95 3.0 0.61 0.88 22.6 12 4 0.68 0.75 0.85 4.8 2.85 2.0 0.53 0.8 21.8 11.1 6 0.6 0.7 0.8 4.1 2.45 1.6 0.44 0.71 19.5 8.1 3 2.5 Extinction coefficient \\(1/cm\\) From experiment at 1st hour 2 From experiment at 4th hour From experiment at 6th hour 1.5 From proposed model at 1st hour From proposed model at 4th hour From proposed model at 6th hour 1 0.5 0 200 300 400 500 600 700 \\[800 900 1000 1100 Wavelength \\\(nm\\\) Figure 4. 18: Extinction coefficient of 0.03 %v/v Alumina nanofluids at different time 6.0 5.0 Extinction coefficient \\\(1/cm\\\) From experiment at 1st hour 4.0 From experiment at 4th hour From experiment at 6th hour 3.0 From proposed model at 1st hour From proposed model at 4th hour 2.0 From proposed model at 6th hour 1.0 0.0 200 300 400 500 600 700 \\\[800 900 1000 1100 Wavelength \\\\(nm\\\\) Figure 4. 19: Extinction coefficient of 0.05 %v/v Alumina nanofluids at different time 12.0 10.0 Extinction coefficient \\\\(1/cm\\\\) From experiment at 1st hour 8.0 From experiment at 4th hour From experiment at 6th hour 6.0 From proposed model at 1st hour From proposed model at 4th hour 4.0 From proposed model at 6th hour 2.0 0.0 200 300 400 500 600 700 \\\\[800 900 1000 1100 Wavelength \\\\\(nm\\\\\) Figure 4. 20: Extinction coefficient of 0.08 %v/v Alumina nanofluids at different time 10 From experiment at 1st hour From experiment at 4th hour From experiment at 6th hour 8 From proposed model at 1st hour Extinction coefficient \\\\\(1/cm\\\\\) From proposed model at 4th hour 6 From proposed model at 6th hour 4 2 0 600 700 800 \\\\\[900 1000 1100 Wavelength \\\\\\(nm\\\\\\) Figure 4.21: Extinction coefficient of 0.03 %v/v Titania nanofluids at different time 10 8 From experiment at 1st hour Extinction coefficient \\\\\\(1/cm\\\\\\) From experiment at 4th hour From experiment at 6th hour 6 From proposed model at 1st hour From proposed model at 4th hour 4 From proposed model at 6th hour 2 0 600 700 \\\\\\[800 900 1000 1100 Wavelength \\\\\\\(nm\\\\\\\) Figure 4. 22: Extinction coefficient of 0.05 %v/v Titania nanofluids at different time To make the model more effective, an attempt was taken to generate a simplified correlation to obtain the values of both of the constants A and B. The correlations are presented in Table 4.10. Applicability of the correlations was defined on the basis of error analysis which is presented in next chapter. Table 4 .10: Simplified correlations for the constants A and B for the Alumina and Titania nanofluids Alumina Nanofluids Titania Nanofluids Correlation Factor \\\\\\\(applicable for \\\\\\\$\phi \leq 0.08\\\\\\\$ %v/v and \\\\\\\(applicable for \\\\\\\$\phi \leq 0.03\\\\\\\$ %v/v and \\\\\\\$350 \leq \lambda \leq 1100\\\\\\\$ nm\\\\\\\) \\\\\\\$700 \leq \lambda \leq 1100\\\\\\\$ nm\\\\\\\) A 1 . 7569 \\\\\\\$\phi\\\\\\\$ 0 . 2107 - 0 . 04 t 3 . 745 \\\\\\\$\phi\\\\\\\$ 0 .491 - 0 . 0334 t 0 . 58 \\\\\\\$\phi\\\\\\\$ - 0 . 659 - 0 . 34 t 0 . 3399 \\\\\\\$\phi\\\\\\\$ - 1 .211 - 0 . 648 t B **CHAPTER 5: DISCUSSIONS 5.1 Introduction Results of this work suggest that there is a strong relationship** among aggregation, size of nanoparticles, concentration and optical properties of nanofluids. The proposed model which includes all the above mentioned parameters is able to provide satisfactory outcome. This chapter is designed to interpret the results of stability behaviour and optical properties of the Alumina and Titania nanofluids as well as characteristic of the proposed model. Furthermore, applicability of the nanofluids in direct absorption solar collectors is also conferred on the basis of\\\\\\]\\\\\\(#\\\\\\)\\\\\]\\\\\(#\\\\\)\\\\]\\\\(#\\\\)\\\]\\\(#\\\)\\]\\(#\\)\]\(#\)](#)

the acquired results. 5.2 Stability It is difficult to study properties of nanofluids without the study of its aggregation kinematics and stability. Result of the stability analysis has shown that Alumina nanofluids were more stable than Titania nanofluids. Total potential energy between the particles in nanofluids and exponential integrant part in (3.8) has been [shown in Figure 5.1 and Figure 5.2](#) for Alumina and Titania nanofluids respectively. Trends of potential energy curves in the above mentioned figures show strong repulsive nature of Alumina nanoparticles. Alumina nanoparticles were found repulsing each other till their surface inter-distance crossed their diameter. On the other hand, Titania nanoparticles were found repulsive till their surface inter-distance equalled their radius. In a constant pH solvent, zeta potentials for both types of nanoparticles were found decreasing with [an increase in](#) concentration. Decrease [in zeta potential](#) means decrease [in surface charge/](#) ion which is responsible for providing electrostatic repulsive force between the particles. Alumina nanoparticles changed pH of the mixture from 4 to 9, whereas Titania did not react with solvent and remained at pH 4. Change of pH value in resultant solution increased ionic concentration of Alumina nanofluids that results higher electrostatic repulsive force (Hiemenz & Rajagopalan, 1997) in it compared with Titania nanofluids.

2.0E+08 E for 0.03% v/v V for 0.03% v/v E for 0.05% v/v V for 0.05% v/v E for 0.08% v/v 25 V for 0.08% v/v 20 1.5E+08 B(h) eVT s/2 KbT 1.0E+08 5.0E+07 15 10 VT / KbT 5 0.0E+00 0 2.0 2.5 3.0 3.5 4.0 s (=2+(H/a))

Figure 5.1: Curves of potential energy (V) and of the quantity B(h) eVT / KbT s 2 (=E) for the Alumina nanofluids 2.0E+08 E for 0.03% v/v V for 0.03% v/v E for 0.05% v/v V for 0.05% v/v E for 0.08% v/v V for 0.08% v/v 25 20 1.5E+08 B(h) eVT s/2 KbT 1.0E+08 5.0E+07 15 10 VT / KbT 5 0.0E+00 2.0 2.5 3.0 s (=2+(H/a)) 3.5 0 4.0

Figure 5.2: Curves of potential energy (V) and of the quantity B(h) eVTs/2KbT (=E) for the Titania nanofluids As a result Alumina nanofluids were found more stable than the Titania nanofluids. Another reasons of low stability of the Titania nanofluids are its higher mass density (4.260 Kg/m³) and particle diameter of 21 nm compared with Alumina particles having 4.0 Kg/m³ mass density and 13 nm particle diameter. They doubled the sedimentation velocity of the Titania particles compared to Alumina particles [as shown in Table 4. 2 and Table 4.4](#). 5.3 Optical Properties of the Nanofluids Results show that experimental extinction coefficients are higher than that estimated by the RA. Absorption coefficients were observed identical for all the particles sizes of Alumina and Titania [in Figure 4. 6 and Figure 4. 12 respectively](#). Although [a](#) bsorption efficiency is dependent on particle diameter as shown in Equation (3.23), absorption coefficient becomes independent of particle diameter when Equation (3.25) is used to get that. Therefore, on the basis of the RA, it can be said that absorption coefficient varies with concentration of dispersed particles in a medium. Hence, variation in extinction coefficient for a particular concentration was resulted for variation in scattering coefficient. It is evident by Literatures (Hartland, 2011) as well as current analysis that scattering of light increases with an increase in particle size. In reality, particles do not grow in size but particles combine together to form a large particle. Due to collision of particles and clusters/aggregates, aggregates become larger with time (Gruy, 2011) and nature of aggregate's packing depends on their interaction with particles (Adachi & Aoki, 2009). Results show that fractal dimensions increase with concentration. As a result, aggregates are becoming denser for higher concentration. Thus, denser aggregates are behaving like a single particle. For both Alumina and Titania nanofluids, it was observed that aggregate size was above 100 nm and approximately consistent for all the concentrations. From DLS analysis results which have been [presented in Figure 4. 3 and Figure 4. 4](#), it was also [observed that](#) nanofluids were containing a series of aggregates having different sizes and intensity. Increase in size and density increases sedimentation velocity as well as scattering coefficient. However, increase in sedimentation rate and aggregates' density reduces barrier of photons. As intensity of aggregates increases, photons get clear path with less or no interaction with particles or aggregates. As a result transmittance of light increases and/or extinction coefficient decreases with time. Therefore, it can be said that consideration of uniformly distributed single particles inside nanofluids and neglecting aggregation phenomena result very low estimation of extinction coefficients by the classical approaches. Another reason might be the change in the properties of nanomaterial compared with bulk materials (Link & El-Sayed, 2000). Surface of nano/micro sized materials are [somewhat different than](#) the [single crystal](#) surface [because of the](#) presence of defects on nano/submicron sized materials (Franks & Meagher, 2003). In the case of nano-sized oxide particles, surface property changes strongly enough to make them of unprecedented sorption characteristics (Fernández-García & Rodríguez, 2008). Alumina nanoparticles also contains surface hydroxyl groups (Shirai et al., 2006). Being amphoteric, surface of Alumina reacts [with acid and base at](#) any value of pH (Hunter, 2000). Change in pH value in our Alumina samples confirm that some chemical reaction was happened. This chemical reaction modifies surface of Alumina particles. Change in Alumina surface has also been reported by Franks and Gan (2007). They have presented the 001 surface structure of Alumina as shown in Figure 5.3 where it has been mentioned that Oxygen resides on the top of the surface in water and Aluminium resides there in vacuum. Titania also interacts with water molecule and it is able to adsorb Cl as well. Both hydration and adsorption processes modify surface structure of Titania as [shown in Figure 5.4 and Figure 5.5](#). Since surface structure is an important parameter for various properties of nano-materials, impurity or defects on the surface may affect optical properties as well (Chiodo et al., 2010). Refractive index of the nanofluids were both experimentally measured and analytically analysed. In both types of samples refractive index for 0.08 %v/v concentration were found between the refractive index values of other two concentrations. This was happened because of instability of the nanofluids of 0.08 %v/v concentration. Refractive index (bulk materials) of Alumina is lower than Titania and they are 1.77 and 2.91 respectively. Therefore, enhancement of refractive index by Alumina was found lower than Titania in the nanofluids. Again, analytical result does not agree with experimental result of refractive index. Effective medium approach considers the dielectric properties of bulk material instead of nanoparticles. Since electron density of nanoparticles on surface is very high, there is a possibility of having different dielectric constant value for nanoparticles. Therefore, more investigation is still required. Figure 5.3: [Schematics of two types of surface terminations of \(a\) Al₂O₃ 001 surface. A\) Al termination in vacuum and B\) O termination in water. Black and grey spheres represent Al and O atoms. \(Source: Franks and Gan, 2007\) TiO₂\(110\) TiO₂\(100\) Figure 5.4: 2 Single-Crystal Surfaces", Langmuir, 1996.">\[Interaction of water with \\(110\\) 2 Single-Crystal Surfaces", Langmuir, 1996.">\\[and \\\(100\\\) surfaces of TiO₂\\]\\(#\\). \\(Source: Henderson, 1996\\) Figure 5.5: Chlorine adsorption mechanism on TiO₂ surfaces. \\(Source: Diebold et al., 1998\\) 5.4 Proposed Model The proposed model for estimation of extinction coefficient is found very promising for Alumina nanofluids. It can estimate extinction coefficient of Titania nanofluid of very low concentrations \\(\\$\leq 0.03\\$ %v/v\\). To check the applicability of the proposed model, an error analysis has been done. Table 5.1 and Table 5.2 present error encountered using both the proposed model and Rayleigh approach. For the Alumina nanofluids \\(as shown in Table 5.1\\), it was noticed that the error in estimating extinction coefficient of the nanofluids ranged from 97.4-1.9% within 82 the region of 350-1100 nm wavelength. This approach, in the most part of the region, was found low estimating the extinction coefficient. In response to the approach, newly developed and proposed model was found estimating the extinction coefficient with the error ranging from 35.2-0.3% in the range of 350-1100 nm wavelength. For the Titania nanofluids \\(as shown in Table 5.2\\), the Rayleigh approach was found having error of 98.7-67.6% within the range of 650-1100 nm whereas the newly proposed model was found with error of 27.5-0.2% for \\$\phi \leq 0.05\\$ %v/v and 83.9-0.3% for \\$0.05 < \phi \leq 0.08\\$ %v/v. Error analysis report for generalized correlations of A and B \\[are presented in Table 5.3 and Table 5.4\\]\\(#\\) for the Alumina and Titania nanofluids respectively. In the Alumina nanofluids, error limits within 30.1-0.4% for the wavelength range of 350-1100 nm for \\$\phi \leq 0.05\\$ %v/v, whereas it remains within 52-0.2% for \\$\phi = 0.08\\$ %v/v. On the other hand, in the Titania nanofluids, error limits within 16.4-0.7% for the wavelength range of 700-1100 nm for \\$\phi \leq 0.05\\$ %v/v, while it remains within 58.5-3.7% for \\$\phi = 0.08\\$ %v/v. Although the perimeter of observed error in Titania nanofluids are\]\(#\)](#)

seemed to be acceptable in comparison with the error resulted from RA, inconsistency/fluctuation in error type (+/-) confines the applicability of the correlation only for $\phi \leq 0.03$ %v/v within the specified wavelength region. This model is unique in the field of nanofluids for extinction coefficient measurement. Colloidal sedimentation, self-assembly of particles, stability and aggregation kinetics were investigated by many researchers, such as Serrano et al. (2011), Romano and Sciortino (2011), Miszta [et al. \(2011\)](#), Ghadimi [et al. \(2011\)](#), Petosa [et al. \(2010\)](#), Chen and Elimelech (2006), etc. But [effect of aggregation kinetics on thermal conductivity](#) was investigated by Prasher et al. (2006). [To the best of the author's knowledge, this is the first attempt to develop a mathematical model for measuring optical property considering aggregation kinetics.](#) Main use of this model will be in design of direct absorption solar collectors. 83 Table 5.1: Comparison of error in the estimated extinction coefficients of the Alumina nanofluids by the proposed model and the Rayleigh Approach Error (%) encountered for proposed model based estimations; Equation (4.1) λ Time (Hour) (nm) 1 4 6 Concentrations (%v) [0.03](#) [0.05](#) [0.08](#) [0.03](#) [0.05](#) [0.08](#) 0.03 0.05 0.08 650 -13.2 -27.5 -83.9 -10.2 -21.1 -79.2 -8.2 -19.5 -75.8 700 -7.1 -11.1 -40.0 -4.5 -7.6 -36.5 -2.6 -6.1 -33.9 750 -2.9 -3.8 -15.4 -0.9 -2.2 -12.8 1.0 -0.7 -10.8 800 0.7 1.0 -4.9 2.3 0.3 -3.4 4.0 1.8 -1.4 850 2.9 3.9 -0.3 4.0 1.3 0.2 5.8 2.9 2.1 900 4.4 5.9 2.1 4.9 2.9 2.3 6.7 4.2 4.2 950 0.4 2.4 -0.5 2.2 -1.0 -0.6 5.2 1.3 2.1 1000 -1.2 0.6 -1.9 0.2 -3.7 -2.1 3.9 -0.5 1.1 1050 4.7 7.6 3.7 3.8 0.7 2.3 6.3 3.2 4.9 1100 4.9 8.0 3.7 3.8 0.3 1.8 6.6 3.1 4.4 [Negative value means "high" estimation](#); [positive value means "low" estimation](#) Error (%) encountered for Rayleigh Approach based estimations 0.03 0.05 0.08 92.5 95.4 97.4 89.5 93.6 96.6 86.0 91.5 95.7 81.7 89.0 94.6 75.6 85.5 93.0 67.4 81.1 91.1 56.8 75.3 88.4 42.8 67.5 85.0 18.6 52.0 76.9 1.9 42.0 72.9 -18.9 23.9 61.9 -29.1 7.5 49.5 -10.7 -0.8 20.7 -12.9 -4.8 10.8 -36.8 -17.4 13.3 -32.7 -18.3 7.8 Table 5.2: Comparison of error in the estimated extinction coefficients of the Titania nanofluids by the proposed model and the Rayleigh Approach Error (%) encountered for proposed model based estimations; Equation (4.1) λ Time (Hour) (nm) 1 4 6 Concentrations (%v) [0.03](#) [0.05](#) [0.08](#) [0.03](#) [0.05](#) [0.08](#) [0.03](#) [0.05](#) [0.08](#) 650 -13.2 -27.5 -83.9 -10.2 -21.1 -79.2 -8.2 -19.5 -75.8 700 -7.1 -11.1 -40.0 -4.5 -7.6 -36.5 -2.6 -6.1 -33.9 750 -2.9 -3.8 -15.4 -0.9 -2.2 -12.8 1.0 -0.7 -10.8 800 0.7 1.0 -4.9 2.3 0.3 -3.4 4.0 1.8 -1.4 850 2.9 3.9 -0.3 4.0 1.3 0.2 5.8 2.9 2.1 900 4.4 5.9 2.1 4.9 2.9 2.3 6.7 4.2 4.2 950 0.4 2.4 -0.5 2.2 -1.0 -0.6 5.2 1.3 2.1 1000 -1.2 0.6 -1.9 0.2 -3.7 -2.1 3.9 -0.5 1.1 1050 4.7 7.6 3.7 3.8 0.7 2.3 6.3 3.2 4.9 1100 4.9 8.0 3.7 3.8 0.3 1.8 6.6 3.1 4.4 [Negative value means "high" estimation](#); [positive value means "low" estimation](#) Error (%) encountered for Rayleigh Approach based estimations 0.03 0.05 0.08 98.3 98.7 98.6 97.8 98.5 98.7 96.6 97.7 98.4 96.2 97.4 98.3 94.3 96.2 97.6 91.9 94.6 96.6 76.9 83.9 89.5 67.6 76.6 84.5 78.0 84.8 90.2 73.1 81.0 87.6 Table 5.3: Comparison of error in the estimated extinction coefficients of the Alumina nanofluids by the proposed model with simplified correlations of A & B Error (%) encountered for proposed model based estimations; Equation (4.1) Time (Hour) λ (nm) 1 4 6 Concentrations (%v) [0.03](#) [0.05](#) [0.08](#) [0.03](#) [0.05](#) [0.08](#) [0.03](#) [0.05](#) [0.08](#) 350 -3.4 -14.6 -4.9 12.8 -16.3 3.7 17.9 -4.5 32.0 400 5.4 -4.3 7.8 19.0 -6.6 15.4 23.4 3.5 39.5 450 13.4 6.2 20.4 24.4 3.1 25.7 28.6 11.5 46.4 500 17.4 13.2 27.4 26.0 8.7 30.9 30.1 15.8 49.8 550 18.8 18.0 32.2 24.5 11.6 33.9 28.8 17.1 51.5 600 18.3 21.3 35.8 19.8 12.9 35.6 24.1 16.9 52.0 650 16.5 22.4 38.4 13.3 12.2 36.0 17.9 14.4 51.4 700 13.4 24.1 40.2 4.3 10.4 35.3 9.2 9.8 50.0 750 8.1 23.5 38.5 -4.0 6.8 31.3 -0.7 3.6 45.2 800 3.5 24.9 39.6 -19.2 2.6 28.8 -13.4 -6.0 41.9 850 -1.8 23.2 36.2 -25.3 -1.8 22.2 -19.0 -13.2 34.6 900 -5.3 17.2 32.0 -29.6 -7.0 13.9 -17.6 -19.2 25.9 950 -1.9 0.8 7.7 4.2 -3.6 -0.2 13.5 -0.9 12.6 1000 -1.7 -2.8 0.3 4.8 -4.5 -6.8 14.0 -0.4 6.4 1050 -7.9 4.3 14.0 -16.4 -11.8 -8.2 -7.3 -18.3 3.6 1100 -7.4 1.8 10.9 -11.8 -11.2 -10.7 -2.3 -15.7 0.9 [Negative value means "high" estimation](#); [positive value means "low" estimation](#) Table 5.4: Comparison of error in the estimated extinction coefficients of the Titania nanofluids by the proposed model with simplified correlations of A & B Error (%) encountered for proposed model based estimations; Equation (4.1) λ (nm) 1 Time (Hour) 4 6 Concentrations (%v) [0.03](#) [0.05](#) [0.08](#) [0.03](#) [0.05](#) [0.08](#) 700 -9.5 -12.3 -51.3 -1.5 1.2 -9.6 -4.6 -16.4 -58.5 750 -5.3 -4.9 -24.7 2.1 6.1 9.3 -0.9 -10.5 -31.0 800 -1.5 0.0 -13.3 5.1 8.4 16.8 2.2 -7.6 -19.9 850 0.7 3.0 -8.3 6.7 9.4 19.5 4.0 -6.3 -15.7 900 2.2 5.1 -5.7 7.5 10.9 21.0 4.8 -4.6 -13.1 950 -2.0 2.1 -8.3 4.6 7.4 17.4 3.0 -6.5 15.0 1000 -3.8 0.8 -9.7 2.5 4.9 15.2 1.5 -7.7 -15.7 1050 2.4 7.3 -3.8 6.2 8.9 19.9 4.1 -4.5 -11.7 1100 2.5 7.9 -3.7 6.0 8.6 19.0 4.3 -4.2 -12.0 [Negative value means "high" estimation](#); [positive value means "low" estimation](#) CHAPTER 6: CONCLUSIONS AND RECOMMENDATIONS 6.1 Conclusions The study was set out to explore the aggregation and optical behaviour of Alumina and Titania nanofluids. Experiments were conducted to measure physical, electrochemical and optical properties. Both experimental and analytical analysis were combined to obtain a mathematical model to be able to estimate extinction coefficient of nanofluids accurately. In response to the decline of fossil fuel reserve, increase in its price, escalation in GHG emission and pollution of environment, it is found very important to harvest green energy to fulfil the rapidly rising energy demand. DASCs with nanofluids are found very effective in cultivating solar energy by many researchers. However, inadequate experimental optical data as well as curbs in classical approaches for metal oxide nanofluids restrict to evaluate and modify the performance of DASCs. Therefore, this study focuses on optical properties of two popular nanofluids such as, Alumina and Titania, which have shown promising and unique behaviour in thermo-physical properties. The study also relates aggregation phenomena with optical parameters to provide a model to calculate extinction coefficient of the nanofluids. In particular, the study was divided in three parts to reach the goal. First of all, stability of the nanofluids was investigated combining experimentally obtained physical properties, such as pH, zeta potential, DLS size analysis report and TEM images, with analytical model for aggregation kinetics. Nanofluids of three concentrations were investigated. Results of this part showed that Alumina nanofluids were more stable compared to Titania nanofluids. Maximum stability ratio was found for 0.03 %v/v Alumina nanofluid which was 2.37×108 . Corresponding time constant of the nanofluid was 41 hours. Repulsive force between Alumina nanoparticles was dominating and it was 86 found active for inter-surface distance of 13 nm (equals particle diameter). In contrast, repulsive force was active for inter-surface distance of 10 nm (equals particle radius) for Titania nanofluids. pH value of Alumina nanofluids was quickly increased from 4 to 9 indicating its reactivity with solvent. On the other hand, pH value of Titania nanofluids was unchanged. Depending on the results of DLS analysis at different time after preparation of nanofluids, fractal nature of aggregates was established. It was discovered that fractal dimension of aggregates was changing with concentration. At very low concentration, aggregates behaved as very porous and they became denser with increase in concentration. Finally, results of this part can be combined to state that stability and aggregation characteristics depend on properties of both particles and basefluids; functionalization process depends on interrelation among particles, basefluid and functionalizing agent. In the second part, extinction coefficients of Titania nanofluids were found higher than Alumina nanofluids for wide range of wavelengths. Reductions in extinction coefficients were also lower than Alumina nanofluids. Up to 44% reduction in extinction coefficient was found for 0.05 %v/v Alumina nanofluid in 400-900 nm within 6 hours. However, reduction in extinction coefficient of Titania nanofluids remained below 10% in visible region and crossed 10% in IR region. Enhancement in refractive index of water was obtained higher for the presence of Titania particles. It can be concluded that the Titania nanofluids exhibited promising optical characteristics. Results of first two parts were combined and analysed together in the last part to express the whole phenomena mathematically. Results showed that the proposed model is able to estimate extinction coefficient of nanofluids more accurately than classic approaches. It was found applicable for the Alumina nanofluids over a wavelength range of 350-1100 nm for concentration up to 0.08 %v/v. In this range maximum encountered 87 error range was found between 35.2-0.3% of experimental values. On the other hand,

the model was found applicable for Titania nanofluids over a wavelength range of 650-1100 nm for concentration up to 0.05 %v/v where the error range was found 27.5- 0.2% of experimental extinction coefficient. Looking at the results of all the above three parts as a whole, it can be concluded that Alumina nanofluids have comparatively good stability at very low concentration, whereas Titania nanofluids have good optical properties. The proposed model is able to provide good result of extinction coefficients of the nanofluids at low concentrations comparative to other approaches.

6.2 Recommendations

Alumina and Titania significantly enhance optical behaviour of water and thus can be used in DASCs. However, use of Titania will be more favourable in the sense of optical enhancement but stability should be improved. Although the proposed model is applicable for Alumina and Titania nanofluids at low concentrations. This type of investigation for different types of nanofluids will certainly help in design of DASCs. As light absorption also affects the surface tension of capillary nanofluids, this type of study may open new door for precision switching systems.

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Appendix A Table A.1: Viscosity of water at different temperatures °C η(cp) °C η(cp) °C η(cp) °C η(cp) °C η(cp) 0 1.7870 21 0.9779 41 0.6408 61 0.4596 81 0.3503 1 1.728 22 0.9548 42 0.6291 62 0.4528 82 0.3460 2 1.671 23 0.9325 43 0.6178 63 0.4462 83 0.3418 3 1.618 24 0.9111 44 0.6067 64 0.4398 84 0.3377 4 1.567 25 0.8904 45 0.5960 65 0.4335 85 0.3337 5 1.519 26 0.8705 46 0.5856 66 0.4273 86 0.3297 6 1.472 27 0.8513 47 0.5755 67 0.4213 87 0.3259 7 1.428 28 0.8327 48 0.5656 68 0.4155 88 0.3221 8 1.386 29 0.8148 49 0.5561 69 0.4098 89 0.3184 9 1.346 30 0.7975 50 0.5468 70 0.4042 90 0.3147 10 1.307 31 0.7808 51 0.5378 71 0.3987 91 0.3111 11 1.271 32 0.7647 52 0.5290 72 0.3934 92 0.3076 12 1.235 33 0.7491 53 0.5204 73 0.3882 93 0.3042 13 1.202 34 0.7340 54 0.5121 74 0.3831 94 0.3008 14 1.169 35 0.7194 55 0.5040 75 0.3781 95 0.2975 15 1.139 36 0.7052 56 0.4961 76 0.3732 96 0.2942 16 1.109 37 0.6915 57 0.4884 77 0.3684 97 0.2911 17 1.081 38 0.6783 58 0.4809 78 0.3638 98 0.2879 18 1.053 39 0.6654 59 0.4736 79 0.3592 99 0.2848 19 1.027 40 0.6529 60 0.4665 80 0.3547 100 0.2818 20 1.002 (Robert C. Weast et al., 1988) Conc. (%v/v) λ(nm) 2.00E-07 2.50E-07 3.00E-07 3.50E-07 4.00E-07 4.50E-07 5.00E-07 5.50E-07 6.00E-07 6.50E-07 7.00E-07 7.50E-07 8.00E-07 8.50E-07 9.00E-07 9.50E-07 1.00E-06 1.05E-06 1.10E-06 Table A.2: Experimental transmittance of Alumina nanofluids at different time periods after preparation After 1 Hour After 2 Hours After 4 Hours After 6 Hours 0.03% 0.23 1.43 9.26 23.42 38.91 51.36 61.76

69.77 75.99 80.53 83.86 84.70 87.30 87.21 86.59 69.46 62.95 80.42 78.91 0.05% 0.09 0.14 1.77 8.26 20.05
32.25 43.81 53.55 61.58 68.18 72.77 74.50 77.73 78.04 78.70 63.75 58.17 74.38 73.40 0.08% 10.37 19.07
28.70 38.12 46.53 53.66 58.45 63.75 66.46 68.23 56.13 51.98 67.39 66.84 0.07 0.05 0.12 0.89 4.41 0.03% 0.25
1.48 9.58 24.25 40.23 53.00 63.68 71.94 78.46 83.14 86.56 87.40 90.06 89.94 89.37 71.45 64.94 82.97 81.32
0.0005 0.1050 0.1540 1.8260 8.4550 20.4630 32.9880 45.0760 55.5070 63.9590 70.5990 75.6780 77.9630
81.5920 82.4150 82.5290 66.3780 60.4900 77.6460 73.55 76.2800 72.98 0.08% 0.07 0.05 0.12 0.96 4.63 11.07
20.43 30.85 41.08 50.21 58.00 63.23 69.07 72.09 74.20 60.84 56.60 0.03% 0.25 1.47 9.60 24.50 40.60 53.53
64.34 72.75 79.42 84.17 87.65 88.52 91.24 91.12 90.66 72.16 65.93 84.37 82.55 0.05% 0.11 0.16 1.90 8.78
21.16 34.00 46.26 56.78 65.31 72.09 77.24 79.54 83.28 84.03 84.31 67.79 61.95 79.57 78.22 0.08% 0.07 0.05
0.12 0.98 4.70 11.30 20.92 31.67 42.25 51.69 59.72 65.09 71.11 74.22 76.43 62.42 58.22 75.74 75.04 0.03%
0.24 1.46 9.57 24.34 40.65 53.64 64.52 72.95 79.70 84.47 87.96 88.83 91.58 91.46 91.02 72.31 66.18 84.76
82.88 0.05% 0.11 0.16 1.99 9.16 21.98 35.22 47.82 58.63 67.38 74.31 79.68 82.19 86.14 87.02 87.25 70.03
64.10 82.47 81.00 0.08% 0.08 0.06 0.13 0.98 4.82 11.58 21.42 32.33 43.13 52.79 60.95 66.41 72.57 75.70
77.87 63.55 59.28 77.01 76.28 Transmittance of Alumina nanofluids (%) Transmittance of Basefluid (H2O+HCl)
0.66 0.85 0.89 0.91 0.91 0.92 0.92 0.93 0.93 0.93 0.93 0.91 0.92 0.90 0.88 0.70 0.63 0.80 0.78 98 Table A.3:
Experimental transmittance of Titania nanofluids at different time periods after preparation Conc. (%v/v)
Transmittance of titania nanofluids (%) Transmittance of Basefluid After 1 Hour After 2 Hours After 4 Hours After 6
Hours (H2O+HCl) λ (nm) 0. 03 0. 05 0. 08 0. 03 0. 05 0. 08 0. 03 0. 05 0. 08 0.03 0. 05 0. 08 200 0.009 0.005
0.001 0.009 0.005 0.003 0.009 0.005 0.007 0.009 0.006 0.009 0.66 250 0.005 0.005 0.002 0.004 0.005 0.004
0.004 0.005 0.003 0.005 0.006 0.005 0.85 300 0.010 0.011 0.004 0.011 0.011 0.007 0.013 0.012 0.008 0.011
0.013 0.009 0.89 350 0.007 0.006 0.003 0.006 0.006 0.004 0.007 0.006 0.005 0.008 0.007 0.005 0.91 400 0.009
0.003 0.002 0.010 0.003 0.002 0.010 0.003 0.002 0.011 0.004 0.002 0.91 450 0.019 0.011 0.008 0.020 0.012
0.009 0.020 0.013 0.008 0.020 0.014 0.008 0.92 500 0.026 0.015 0.010 0.027 0.016 0.013 0.027 0.018 0.013
0.027 0.019 0.013 0.92 550 0.041 0.021 0.013 0.042 0.021 0.016 0.043 0.024 0.016 0.043 0.025 0.016 0.93 600
0.142 0.028 0.017 0.145 0.028 0.021 0.148 0.034 0.021 0.149 0.034 0.021 0.93 650 0.610 0.054 0.022 0.627
0.054 0.027 0.641 0.066 0.027 0.645 0.066 0.027 0.93 700 1.952 0.185 0.032 2.006 0.190 0.039 2.055 0.241
0.039 2.072 0.246 0.039 0.93 750 4.459 0.617 0.064 4.586 0.640 0.078 4.705 0.830 0.078 4.747 0.841 0.079
0.91 800 8.439 1.687 0.207 8.681 1.756 0.256 8.918 2.337 0.258 9.003 2.357 0.259 0.92 850 13.279 3.572
0.614 13.667 3.716 0.766 14.048 4.905 0.773 14.182 4.979 0.778 0.90 900 18.685 6.340 1.509 19.242 6.598
1.822 19.818 8.444 1.844 20.024 8.445 1.856 0.88 950 19.671 7.959 2.481 20.242 8.318 2.965 20.708 10.604
2.984 20.909 10.451 3.013 0.70 1000 21.906 10.259 3.957 22.574 10.708 4.617 23.277 13.346 4.674 23.541
13.373 4.710 0.63 1050 32.923 17.320 7.911 33.925 18.026 9.131 35.097 22.179 9.260 35.505 22.158 9.326
0.80 1100 36.569 20.995 10.996 37.697 21.855 12.667 38.928 26.724 12.777 39.354 26.478 12.894 0.78 99 2 4
5 6 10 13 16 17 19 21 22 25 26 29 32 33 34 36 37 38 39 40 41 42 43 44 45 47 48 49 50 51 52 53 54 56 57 61
62 63 64 65 66 69 70 71 72 73 74 75 76 77 78 80 81 84 85 88 89 90 91 92 93 94 95 96 97