LASER LIGHT SCATTERING FOR INVESTIGATION OF PARTICLE SIZE DEPENDENCY OF QUALITY AND FLAVOR OF COFFEE

By

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Abstract

In Ethiopia, the methods of coffee variety and standard identification is often through traditional inspection and previous experience which is subjective and non efficient with the basis of attributes of color, size, shape and flavor, frequently examined by human inspectors. In this research, efficient and objective discrimination of coffee varieties and quality determination method is developed by investigating particle size dependency of quality and flavor of coffee using Laser light scattering technique. Particle size determination for five types of coffee samples (Unwashed Harar-4, Washed Lekempti-2, Washed Sidamo-2, Washed Yirgacheffee-2 and Washed Limu-2) were performed after determination of their optical properties with three phases of experiments. The results show that, all tested samples has high scattering coefficient in the forward direction as compared to their negligible absorption coefficient. And their scattering coefficient has direct relation with their particle size as well as their quality and flavor, the larger the scattering coefficient is the finest the particle size and the highest the quality with good flavor. Accordingly, Washed Yirgacheffee-2 has highest scattering coefficient and finest particle size with succession of others. In line with this, using liquid-liquid caffeine extraction method the participation of caffeine on scattering was tested for Unwashed Harar-4 Coffee sample. And the result show that, caffeine has its own comparable effect on scattering. In general the obtained results were in excellent agreement with those obtained using traditional cup-test and other known methods. Thus, since the developed method have wider economic and scientific applications, even for other turbid mediums, light scattering method is the most preferable for classification of coffee quality standards.

Key Words: Coffee, Standard, Airy Disk, Airy Pattern, Extraction, Scattering Optical Properties, Particle Size

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Introduction

Quality is a degree of excellence in which something distinguishably attributed. It may depend on different factors, in which how and what measurements of an object/matter take place. Different parameters can affect the quality of coffee in different cultivation area of the given country.

Amongst various types of coffee cultivated in the world, it is believed that only qualified Arabica coffee is originated and brewed in different regions of Ethiopian high lands. Ethiopia is not only the icon of coffee, but it thrives on coffee production and people drink coffee regularly in every part of the country. Coffee is closely associated with the Ethiopian culture. Most people in the country start their day by taking a cup or two of coffee in the morning. And also Coffee ceremony, the tradition of serving coffee in Ethiopia is unique.

Coffee quality differs to everyone depending on their upbringing as well as their society. An Ethiopian believes quality coffee should have a good flavor (the simultaneous sensation of aroma and taste), and after being brewed in the traditional clay pot, is drank steaming hot and unsweetened, although some prefer sugar or even a dash of salt.

External and internal factors can contribute to the quality of coffee beverage. Mainly man and nature are external factors. Natural phenomenon include altitude, duration and severity of rainfall, type of soil, pH, genetic origin, location of producing area, chemicals and pesticides, harvesting methods and timing, packing and so forth[1]. Chemical compositions of coffee like caffeine chlorogenic acids and other aromatic and active components are also an internal factor that contributes to the quality and flavor of coffee beverage.

Coffee production for international market in the country goes through many processes in order to be competitive to the world market. Due to this, the government has given serious monitoring and care to preserve the inherent coffee quality characteristics to satisfy customer preferences. Accordingly, in Ethiopia, every coffee produced has to go through the monitoring of the institute of Ethiopian Coffee Quality Inspection Center, in Addis Ababa or Dire Dawa, to certify that the supplied coffee has met at least the minimum requirement of national standard for export [2,3,4].

Processing coffee is the method of converting the raw coffee fruit (cherry) into the commodity. In Ethiopia, there are two ways of coffee processing. They are wet method (washed coffee) and dry method (natural coffee). The coffee supplying areas for washed and unwashed coffee include Yirgacheffe, Sidamo, Limu and Bebeka. Mainly unwashed coffees are from Harar, Jimma, Bale, Wellega Nekempte and Illubabor. Coffee in each area has specific physical and chemical properties which attributes to distinct characteristics of the region.

Coffee is graded for export with the objective of producing the best cup quality and there by securing the best price possible. However, there is no universal grading system. Each producing country has their own national standard which fulfills the minimum export quality requirement suggested by the market[2,3,5]. Most probably the flavor and quality of coffee depends on the coffee beans selected, the roasting process, the brewing method and the fineness of the ground coffee used.

Each brewing method will brew better if the beans are ground to the optimal size in the first place. The objective of the grinding is the reduction of the size of the roasted bean to facilitate the extraction of the flavor and of the soluble compounds during the preparation of the beverage. The time of contact between water and coffee determines how much flavoring material of the coffee could be extracted from the ground coffee. To avoid a low development or an excess of extraction of the components of the flavor, one should use the correct size of the particles of the ground coffee. This is why it is necessary to adjust the grinders to guarantee this effect. In commercial grinding, the process is well developed and monitored by the use of laser diffraction particle size analyzers. The speed, consistency, and ease of use have made it the most popular method for process control[6].

In Ethiopia, coffee grading is conducted through the combination of two methods [3]. They are green coffee (raw bean) analysis and cup tests (liquoring). Green coffee analysis involves visual inspection of physical characteristics of coffee bean. This includes screen analysis which makes size assessment, defect count, appearance or color test and shape which usually refers to the structure of beans. Cup test is based on roasted ground coffee analysis (chemical process) by which aroma, acidity, and other flavor components are tested. From the overall grading methods of coffee, green analysis accounts 40% and cup test accounts 60% in the quality inspection processes.

Coffee variety identification is very useful in encouraging good quality coffee production, ensuring dependable and competent exporters as well as creating lasting business relationship with overseas clients. In Ethiopia, the method of coffee variety identification is through traditional inspection and previous experiences which is subjective and non efficient. The basis of coffee variety identification is often subjective with attributes color, size, shape and flavor frequently examined by human inspectors[2,5]. It is also found that human perception could easily be biased. As a result, objective discrimination of coffee varieties and quality determination is necessary which is consistent, non-destructive and cost effective for commercial purposes.

In light of these, it is useful to explore the possibilities of adopting the most economical and faster system which saves time and is more accurate in classification of quality of coffee by reducing observer effects of biases pertaining to the quality standard that enhances the commercial needs. One of such best method is scattering of laser light using roasted, grounded and dissolved coffee solutions.

Scattering experiment is an applied spectroscopic technique that used to study the physical system when it interacts with electromagnetic radiation. More over it is a spectroscopic technique that studies about the production, transmission and detection of light with relation to the optical properties of a material medium.

In this thesis, we used laser light scattering experiment to investigate optical properties of variety of coffee samples, as well as investigation of particle size dependency of coffee flavor and quality. It has been so difficult for coffee quality assurance organizations and coffee exporters in the country as well as for quality control laboratories to determine particle size dependence of quality of coffee rapidly, precisely and with less labor cost. Most quality control laboratories widely used physical and chemical analysis methods in which green coffee (raw bean) and cup test (liquoring) analysis respectively for identification of coffee quality. However, these physical and chemical methods for identification of coffee quality are expensive, time and labor consuming and less precise.

For many years measurement techniques based on laser light scattering theory have been applied to obtain information about the size, structure, shape, composition and concentration of substances[7,8]. In this research, laser light scattering experiment is carried out in the following manner. That is, scattered light intensity (at different scattering angle) and transmitted light (at 0^o angle) were measured experimentally and the result was calculated and analyzed by using Beer-Lamberts law and radiation transfer equation(RTE) to determine the optical properties of variety of coffee samples, and then using angular distribution scattering intensity measurements, particle size determination was performed.

Therefore, this research work will initiate a model for coffee variety classification which is consistent, efficient, time saving, cost effective and direct by exploring the technology of laser light scattering through the scattering coefficient and particle size parameter analysis. More over it is simple, sensitive, producible, valid and the most suitable for on line monitoring.

Objectives of the Study

The main objective of this research is to investigate the dependency of quality and flavor of coffee on particle size using laser light scattering experiment technique.

In light of this general theme, the specific objectives of the research are the following.

- ✓ To introduce a model of direct ease and precise method for coffee quality classification and grading system;
- ✓ To introduce a method of measuring quantitative absorbance of a specific coffee sample solution by collimated transmission intensity measurement;
- ✓ To determine the optical properties of the sample such as absorption, scattering and extinction coefficients; and anisotropic factor for different concentrations of the sample under the study (roasted grounded coffee);
- ✓ To measure the intensity of the transmitted light through the sample;
- ✓ To measure the variation of the fluency rate inside the diffusing medium by variable concentration method; and
- ✓ To determine the scattering particles size by angular distribution of intensity measurement.

Organization Of The Thesis

In chapter-1 theory of electromagnetic radiation and its interaction with isotropic dielectric material medium is discussed briefly. In this chapter Properties of electromagnetic radiation, Lasers and their properties and applications, light propagation theories like transmission, absorption, reflection and scattering of light with their respective quantitative and qualitative definitions as Beer-Lamberts law, Snells law and Fresnels laws are presented.

In chapter-2 Model of light propagation through isotropic dielectric material medium is presented. Here radiation transport equation (RTE), methods of solving RTE in which analytical method of diffusion approximation equation and its assumption is discussed in detail. Solution of Diffusion approximation equation for infinite medium and fluency rate measurement equation are also presented in this chapter.

In chapter-3 Materials and methods used in this work are presented. This chapter has two sections. The first section of this chapter deals about the description of different chemicals, samples of coffee types and instruments used to carry out this research. The second section of this chapter deals with methods of sample preparation, method of caffeine extraction for particle size measurement of coffee without caffeine and optical measurement techniques of three phases of the experiment in which collimated transmission, fluency rate and angular distribution intensity measurements are presented.

In chapter-4 The Results and Discussions of the experiment are presented. Tabular expression of results, discussion and analysis of the result using theoretical models and formulas are presented. In this chapter results will be calculated using tables and graphs. In this chapter the results will be compared with known and traditional identification method values. and again here particle size measurement results of coffee sample solution and the caffeine extracted coffee sample solution will compare with each other to know the participation of caffeine for scattering of light.

In chapter-5 Conclusion and Recommendation for future outlooks of the research will be presented.

Finally, Reference materials used in this research will be reported as bibliography.

Chapter 1

Theory of Light Propagation In An Isotropic Dielectric Medium

propagation of light through material medium is the most important and interesting aspect of light. In this chapter the theories of interactions of light with matter are discussed. Starting from the definition and properties of electromagnetic radiation this chapter presents how and what phenomenon take place when light and matter interacts. More over it deals about mathematical representations that can often be used for qualitative and quantitative expressions of varied optical phenomenon exhibited by medium like reflection,absorption and scattering of light.

1.1 Electromagnetic Radiation

Electromagnetic radiation (EMR) is radiant energy that travels outward in all directions from its source through space with or without the need of a material medium. There are different types of EMR, some very familiar, and others rarely mentioned outside scientific discussions. Electromagnetic radiation includes AM/FM radio waves, microwaves, visible light (red to violet), ultraviolet radiation, χ-rays, and gamma rays. Light is a form of electromagnetic radiation in the visible range from its broadened spectrum[9].

Electromagnetic radiation types are identified based on their frequency, wavelength, and source. The energy sources that produce EMR vary greatly, from nuclear reactions in the Sun, which generate gamma radiation, to chemical reactions in the human body that generate infrared radiation (heat)[9].

The electromagnetic spectrum is the term applied to all the types of EMR considered together in terms of frequency, wavelength, or energy. All parts of the spectrum are found, with varying intensity, in our natural environment. We are most familiar with the visible spectrum since we sense it directly with our eyes. And it ranges from 390nm to 780nm wavelength. The infrared spectrum in which it's Wavelengths $\lambda > 780nm$ is sensed as heat and the ultraviolet spectrum ($\lambda < 390nm$) includes radiation that can damage living cells, often causing a physiological response such as sun burn. Other parts of the spectrum may be present as background radiation. Natural background radiation originates from two primary sources: cosmic radiation and terrestrial sources. Cosmic radiation from deep space interacts with atoms in the atmosphere producing χ-rays and unstable isotopes. Terrestrial sources of gamma radiation include radioactive isotopes of uranium, radon, potassium, and carbon[9]. The relative energy of the different types of EMR varies with frequency across the spectrum. here below figure(1.1) shows the distribution of electromagnetic radiation spectrum.

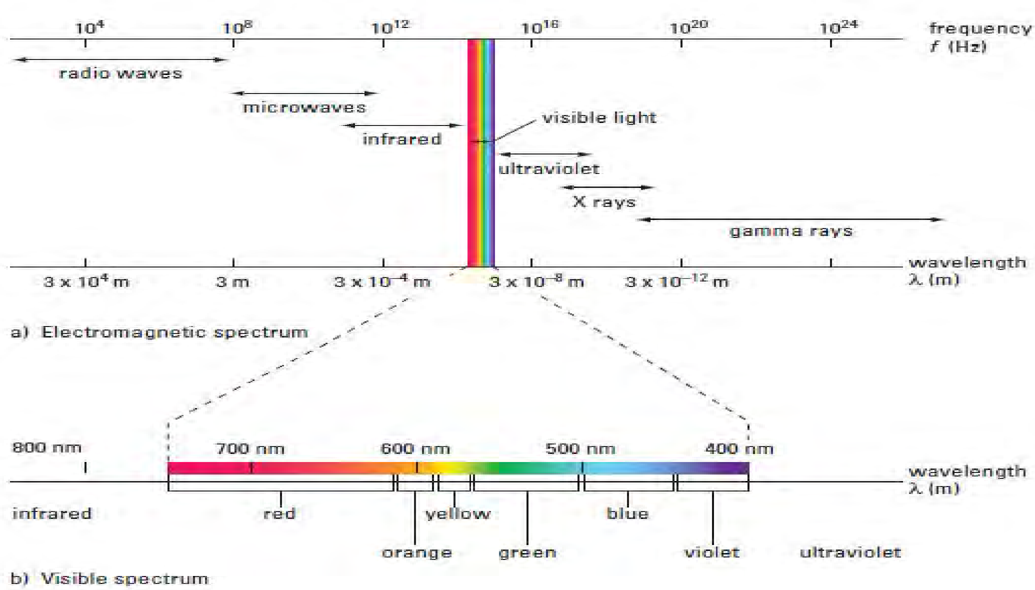


Figure 1.1: *the electromagnetic spectrum showing the visible range.*[9]

The actual light wave may be treated as a collection of plane monochromatic polarized waves of various frequencies. It may be more correct physically to represent an actual light wave as stream of micro particles, called photons[10]. Depending on the former nature of light, propagation of Electromagnetic Radiation or light through the material medium can be described by Helmoltz wave equation derived from four Maxwells equations.

The dual natures of light categorize it's properties as wave like properties, (interference, polarization etc) and particles like properties, (photoelectric effect, Compton effect etc). The study of EMR specifically light propagation through material medium needs special properties of light which can be maintained constant or variable depending on the objective and the method of study.

1.2 Laser and Laser Applications

Most spectroscopic measurement techniques use a special light which has special properties for special conditions. The most known special light is laser light. The word LASER is an acronym for the most significant feature of laser action: Light Amplification by Stimulated Emission of Radiation. There are many different kinds of lasers, but they all share a crucial element: Each contains material capable of amplifying radiation. Thus Laser is a device that produces a beam of intense, directional (linearly polarized) and monochromatic light in which all the waves are in phase or are coherent. Lasers have three basic components: laser active medium, energy source (or pumping system), and resonant optical cavity with reflecting mirrors[11]. Active medium: contains atoms or molecules whose electrons may be excited to an elevated energy level by an external energy source (pumping source) to form population inversion. The excitation mechanism functions by pumping energy into the active medium by one or more basic methods such as optical, electrical, chemical, etc. The resonator is composed of high reflectance mirror that reflects essentially 100% of light, and a mirror that allows partial transmission.

Many scientific, military, medical and commercial laser applications have been developed since the invention of laser in 1958. The coherency, high mono-chromaticity, ability to reach extremely high powers with narrow band width, large frequency tuning range over considerable values and all other properties of Laser light allows it to play tremendous specialized applications[12]. The numerous fields of laser applications may be broadly categorized in to two groups.

- One group involves applications where laser beams play a role of high power beam. This is exploited to produce a targeted effect on material. These applications include material processing (e.g., welding, heat treatment, cutting, hole burning, isotopes separation etc.)[11].
- The other group include data transmission and processing, measurements and quality control applications[11].

The high purity/mono-chromaticity of laser makes it as a very useful source for spectroscopy. For many years measurement techniques based on laser light scattering theory have been applied to obtain information about the size, structure, shape, composition and concentration of substances. Thus, for this scattering experiment, a linearly polarized continuous He-Ne laser with 35mw power at 633nm wavelength is used to investigate scattering properties and particle size dependency of quality and flavor of grounded coffee.

1.3 Light Matter Interaction

When a beam of light is incident on material medium and propagate through it, various light-mater interaction phenomenon takes place. These interactions can occur at the surface or inside the medium components and the rest of light may penetrate the medium (transmission). Due to this interaction of light with the medium components there might be reflection on the surface and attenuation due to scattering and absorption inside the medium by internal suspended components[13].

The absorption properties are strongly dependent on the wave length of the incident light while reflection and scattering have negligible effect on the wave length of the incident light. All these processes give concrete information about the size, concentration, motion and shape of the constituents of the medium[7,8]. The information we will obtain from this experimental work has a paramount importance in calculating the optical properties of the medium (coffee) and it's particle size effects on the quality and flavor.

The interaction of light with material medium depends on the composition & concentration of the medium and the wavelength & energy of the light. A solution of coffee is turbid, and suspended coffee particle in the solution may scatter most of light energy in all directions. And it may have a little absorption peak on the corresponding wave length.

In any conditions of material medium,when light tries to propagate through the given medium the following properties of light can happen.

•Reflection and Refraction

•Absorption

•Scattering

Here in figure(1.2) a typical situation is shown, where laser light beam is incident on material medium (specifically coffee solutions for this typical work) and it's interaction inside with suspended particles of coffee.

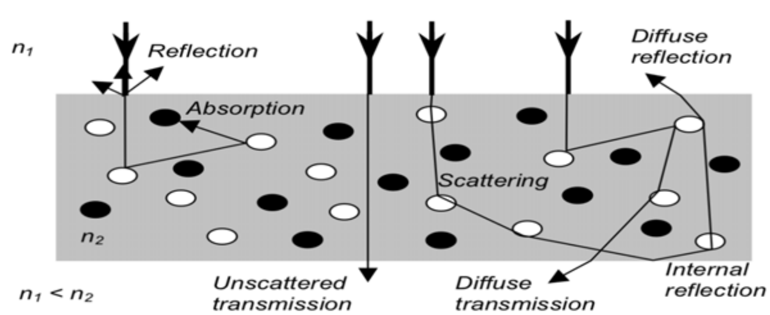


Figure 1.2: *Geometry of Reflection, Refraction, Absorption and scattering*

1.3.1 *Transmission of He-Ne laser beam through coffee sample solution*

When collimated He-Ne laser beam impinges on a sample solution, a fraction of light energy is transmitted through the sample and the rest fraction is losted by three processes. These processes are reflection,absorption and scattering. Figure (1.2) above illustrates the interaction between Laser light and sample medium.

Assume that the total incident light intensity is I_0 , the reflected light intensity is I_r , absorbed light intensity is I_a , transmitted light intensity is I_t and the scattered intensity of light is I_s . According to the conservation principle the normalized total incident intensity of light fulfills the condition;

$$I_0 = I_r + I_a + I_S + I_t \tag{1.3.1}$$

and divide both sides of the above equation by I_0 to write as

$$1 = R + A + T \tag{1.3.2}$$

where R is Reflectance (the ratio of reflected to incident intensities), T is Transmittance (the ratio of transmitted to incident intensities) and A is Absorbance in which the sum total of absorbed and scattered fraction of light intensities.

1.3.2 Reflection and Refraction

Reflection and refraction are the most common phenomenon when light and matter interacts each other. Light intensity will be reflected or/and refracted because of a refractive index mismatch between the air and the sample boundary. The intensity of both reflected or refracted light doesnt only depend on the difference in refractive index mismatch but also depend on polarization, Angle of incidence, wavelength of light and structure & shape of the surface.

Reflection and refraction are strongly related to each other by Fresnel's law. Reflectance or The fraction of reflected intensity R, for unpolarized light is defined as the ratio of the reflected intensity from the medium to the incident intensity. This can be expressed as Fresnel's law[14],

$$R = \frac{1}{2}([\frac{n_1 cos\theta_i - n_2 cos\theta_t}{n_1 cos\theta_i + n_2 cos\theta_t}]^2 + [\frac{n_2 cos\theta_i - n_1 cos\theta_t}{n_2 cos\theta_i + n_1 cos\theta_t}]^2) \tag{1.3.3}$$

where n_1 and n_2 are the refractive indices for the external medium (air) and material respectively. θ_i and θ_t are the incident and the transmitted angle respectively.

using Snell's law of refraction

$$n_1 sin\theta_i = n_2 sin\theta_t \tag{1.3.4}$$

$$\Rightarrow \theta_t = sin^{-1}(\frac{n_1}{n_2} sin\theta_i) \tag{1.3.5}$$

When the light is incident in the direction normal to the medium surface, i.e. $\theta_i = \theta_t = 0^0$, and if the external medium is air ($n_1 = 1$), and $n_2 = n$, equation (1.3.3) becomes

$$R = (\frac{n-1}{n+1})^2 \tag{1.3.6}$$

1.3.3 Absorption

Absorption is due to a partial conversion of light energy into heat, motion or certain vibrations of molecules of the absorbing material. Instead of completely transmitting light, an object or a substance can absorb part or all of the incident light, usually by converting it into heat. A substance is said to show general absorption if it reduces the intensity of all wave lengths of incident light nearly with the same amount. Instead many materials absorb some wavelengths while transmitting others, which is called selective absorption[15]. Laser light is a monochromatic (i.e. single wavelength), which is responsible for its selective effects on the given sample medium.

Absorption occurs when the photon frequency incident on the medium matches with the frequency associated with the molecules transition. The ability of a medium to absorb electromagnetic radiation depends on a number of factors, mainly the electronic constitution of its atoms and molecules, the wavelength of incident radiation, the thickness of the absorbing layer, and the internal parameters such as the temperature and/or concentration of absorbing agents.

The relationship between the absorption of light in a purely absorbing medium of thickness x, can be found using Beer-Lambert law[15]. For plane waves, The fractional reduction of the beam intensity $\frac{dI}{I}$ is directly proportional to an infinitesimal path length dx in it's direction through a homogeneous medium with absorption coefficient μ_a i.e,

$$\frac{dI}{I} = -\mu_a dx \tag{1.3.7}$$

Integrating and evaluating it as definite integral in traversing a finitesimal thickness x we can find

$$\int_{I_0}^{I} \frac{dI}{I} = -\mu_a \int_0^x dx \tag{1.3.8}$$

and the reduced intensity (I) can be written as

$$\Longrightarrow I = I_0 exp(-\mu_a x) \tag{1.3.9}$$

The absorption coefficient μ_a at a particular frequency can be related with the absorption cross-section σ_a and the number density of molecules ρ in the sample as follows

$$\mu_a = \rho\sigma_a \tag{1.3.10}$$

And it can be related again with the concentration c and absorptivity as $\mu_a = c\varepsilon_a$ then using the above two relations, the reduced intensity (I) can be rewritten as

$$I = I_0 exp(-\rho\sigma_a x) \tag{1.3.11}$$

$$I = I_0 exp(-c\varepsilon_a x) \tag{1.3.12}$$

The absorption coefficient μ_a in equations above can thus be interpreted as the probability that the photon will be absorbed by the medium per unit length. The reciprocal $\frac{1}{\mu_a}$ is called the absorption path length and equal to the mean free path a photon travels between successive absorption events. This is the distance required for the intensity (I) of the beam to fall to 1/e of the initial intensity (I_0) value.

Abebe B.[2011] and Tadelech A.[2011] studied the Absorbance properties of components of coffee and tea using UV-Visible spectroscopic techniques. UV-Visible spectroscopy measurements tells us that Molecules in the solution of coffee absorb most of the light within the range of wavelength 200nm to 500nm at room temperature[16]. Absorption properties of coffee are often related to the absorption property of the particular groups of components in it. The major absorbing components of coffee are; water, caffeine, chlorogenic acid and other simple components in the coffee. Water absorbs mostly in the ultraviolet and infrared region, and transmits in the visible portion of the spectrum. these above components of coffee absorb light differently in different types of coffee with the same and different species. It means that green, roasted and roasted grounded arebica coffee has different peaks of absorption wavelength range.

1.3.4 Scattering

The scattering of light may be thought of as the redirection of light that takes place when an electromagnetic (EM) wave (i.e. an incident light ray) encounters an obstacle or non- homogeneity, in our case the scattering particle[17]. It is a fundamental factor in our visual perception of this worlds phenomenon, such as the blue sky, red sunsets, clouds, rainbows, snow, fog, milk, and white paint are striking examples of the influence of scattering.

When a cloud of smoke suspended particles, which has elastically bound charged micro particles, are exposed to electromagnetic waves, the particles are set into motion or vibration by the electric field. Unlike absorption, scattering of light occurs at a non-resonance frequency, in which it takes place at frequencies not corresponding to those natural frequencies of particles. The oscillation or perturbation of the electron cloud on the particle results in a periodic separation of charge within the molecule, which is called an induced dipole moment. The oscillating induced dipole moment is manifest as a source of EM radiation, thereby resulting in scattered light. The majority of light scattered by the particle is emitted at the identical frequency (ν_o) of the incident light, a process referred to as elastic scattering.

Rayleigh and Mie scattering are two types of elastic scattering, while Raman and Brillouin scattering are inelastic scattering in which the frequency of the scattered wave differs from that of incident wave. Rayleigh scattering is applicable to small (smaller as compared to incident wavelength), dielectric (non-absorbing), spherical smoke particles suspended in the solution, where as theory of Mie scattering encompasses the general spherical scattering solution (absorbing or non-absorbing) without a particular bound on particle size, most preferably structures with the same scale to the wavelength of the incident light wave[17].

The criteria for Rayleigh scattering is that $\chi \ll 1$ and $\mid m \mid \chi \ll 1$, where χ is the dimensionless size parameter given by the expression

$$\chi = \frac{2\pi a}{\lambda} \tag{1.3.13}$$

where a is the spherical particle radius, and λ is the relative scattering wavelength defined as

$$\lambda = \frac{\lambda_0}{m_0} \tag{1.3.14}$$

where λ_0 is the incident wavelength in vacuum, m_0 represents the refractive index of the surrounding medium and m is the refractive index of the scattering particle.

Assuming the sample is homogeneous so that the scattering particles are uniformly distributed throughout its entire body. The non-scattered components of light I_x through a non-absorbing sample of thickness x=L, can be related with scattering coefficient μ_s as

$$I_x = I_0 exp(-\mu_s x) \tag{1.3.15}$$

The scattering coefficient μ_s at a particular frequency can be related with the scattering cross-section σ_s and the number density of molecules in the sample as

$$\mu_s = \sigma_s \rho \tag{1.3.16}$$

The scattering path length $1/\mu_s$ is the average distance the photon travels between successive scattering events which is known as mean free path.

When a photon that is incident along a direction described by a unit vector, $\hat{s}$, experiences a scattering event, the angular probability of its being scattered in to the direction, $\hat{s'}$, with an angle θ is given by the normalize phase function, $P(\hat{s} \cdot \hat{s'})$. It can be expressed as a function of the cosine of the scattering angle, $P(\hat{s} \cdot \hat{s'}) = cos\theta$

The mean cosine of the scattering angle called anisotropy factor, g, and it is a measure of degree of scattering which can be expressed as follow[18];

$$g = \langle cos\theta \rangle = \int_{4\pi} cos\theta P(cos\theta) d(cos\theta) \tag{1.3.17}$$

If g is positive, the phase function is predominantly forward scattering; if g is negative, backward scattering dominates. A constant phase function results in an isotropic scattering (g = 0). Hence The transport (or reduced scattering coefficient), μ_s' is defined as

$$\mu_s' = \mu_s(1 - g) \tag{1.3.18}$$

In most turbid medium absorption and scattering may be present simultaneously. Their total attenuation coefficient can be expressed as a combination of scattering and absorption coefficients of the medium by

$$\mu_t = \mu_a + \mu_s \tag{1.3.19}$$

This tends us to express the total mean free optical path of incident photons in the medium as

$$L_t = \frac{1}{\mu_t} = \frac{1}{\mu_a + \mu_s} \tag{1.3.20}$$

In some cases, either μ_a or μ_s may be negligible with respect to each other, but it is important to realize the existence of both processes and the fact that usually both are operating. Also, it is very convenient to define an additional parameter, the optical albedo α, by

$$\alpha = \frac{\mu_s}{\mu_t} = \frac{\mu_s}{\mu_a + \mu_s} \tag{1.3.21}$$

For $\alpha = 0$, attenuation is exclusively due to absorption only, whereas in the case of $\alpha = 1$, only scattering occurs. For $\alpha = 1/2$, the total attenuation coefficient $\mu_t = 2\mu_a = 2\mu_s$ in equation above or/and $\mu_a = \mu_s$ which means the coefficients of absorption and scattering are of the same magnitude. In general, both effects may take place but they may occur in variable ratios[19].

Intensity Distribution of scattering of light and Airy Disk

In most turbid mediums photons are preferably scattered in the forward direction. The intensity of the scattered light is a function of the wavelength λ, the scattering angle θ, the particle size diameter d, and the relative index of refraction n of the particle and the surrounding medium. Symbolically, then, $I_s = I(\lambda, \theta, d, n)$.

During the interaction of laser light with particles of the medium, either the particle has larger or smaller in size as compared to the incident light wavelength, Mie scattering theory describes the phenomenon by considering the optical properties (refraction and absorption) of the particles. The smaller the particles are the higher is the contribution of refraction and absorption to the light scattering pattern. For the measurement of particle size distributions inside the given sample medium, therefore, the scattering intensity distribution pattern should be detected over the entire angle range around the sample, and it will looks like as shown in figure(1.3).

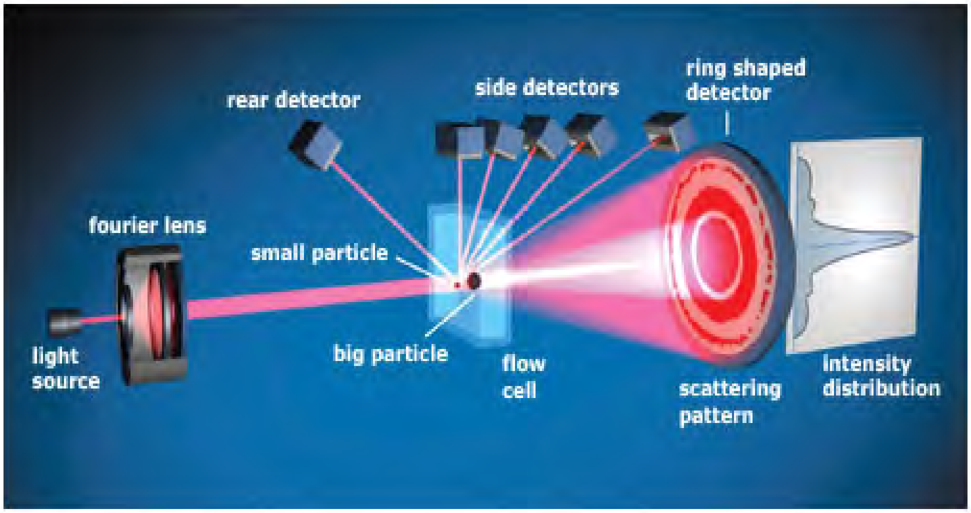


Figure 1.3: *intensity distribution profile of light propagation through medium (airy disk)*[20]

The scattering intensity distribution pattern resulting from a scattering of laser light has a bright region in the center, known as the *Airy disk* which together with the series of concentric bright rings around is called the *Airy pattern.* The dark ring pattern between two bright regions show the detected minimum intensity distribution.

The diameter of the airy disk, d, at the center of the distribution corresponds to the particle size of the scatterer in the medium[15], that is helpful for calculating the dimensionless size parameter χ, which can be expressed using diffraction equation as follows

$$d = \frac{1.22\lambda}{\theta} \tag{1.3.22}$$

where the angle θ measures from the incident transmission line to the first detected minima.

The particle size calculation from the scattering intensity distribution pattern is useful for investigation of the optical properties of the medium specially here coffee medium samples. proper roasting and grinding of coffee (per brewing method) will help us to get quality coffee which can release better flavor. Coffee flavor is composed of the water soluble organic and inorganic natural chemical components of roasted grounded coffee beans, which are extracted as liquids and gas during the brewing process. our sense organs can record the sensations of aroma through olfactory cells located on the organelles. Depending on particle size of roasted grounded coffee, one or mixed testes may be detected at a time. Most researches announce that scattering coefficient calculations for coffee rather than Ethiopian coffee is in the order of $\sim 10^{-2}$[21]. But here in Ethiopia there is no such research which can show the scattering or absorption coefficients of coffee. Where as scattering experiment is done effectively and efficiently for different types milk sample mediums to find the particle size as well as concentrations of fats and proteins. Thus it is necessary to investigate a model for particle size and optical properties of coffee to use its results for classification of standards for export and indoor uses.

Chapter 2

Model of Light Propagation Through Turbid Medium

2.1 Introduction

When beam of light is passed through sample medium, its propagation is affected in two important ways: (1) the intensity will decrease as the light penetrates farther in to the medium, and (2) the velocity will be less in the medium than in free space. The loss of light intensity is chiefly, due to absorption, although under some circumstances scattering may play an important part[15]. In this chapter we shall discuss the model how beam of light propagate through the sample medium (specifically through medium of coffee sample solution for this typical experimental work). The model we used here is Radiative Transfer Equation (RTE), which can be solved analytically with Diffusion Approximation. RTE model is used as good by other researchers for scattering experiment to determine fat and protein concentrations of milk and to investigate optical properties and particle size of milk sample solution with Indian ink absorber. But here for coffee sample solutions, we used variable concentration method which makes the experiment easier and faster. even though Photon transport in turbid medium can be equivalently modeled numerically with Monte Carlo simulations, analytical solution of RTE with diffusion approximation is more efficient computationally.

Thus in this chapter mathematical model of photon transport through turbid medium and it's solution will be presented. More over quantitative and qualitative expressions which can be used for determination of scattering, absorption, reduced scattering coefficients and anisotropy factor of the medium will be discussed in detailed.

2.2 Radiative Transfer Equation (RTE)

The physical phenomenon of energy transfer (photon transfer) in the form of electromagnetic radiation is called Radiative Transfer. The propagation of radiation through medium is affected by absorption, emission and scattering processes. The equation of radiative transfer describes these interactions mathematically.

The fundamental quantity which describes a field of radiation is nowadays called the spectral radiance, traditionally called the specific intensity. The flow of radiation energy through a small area element in the radiation field can be characterized by radiance $L(\vec{r}, \hat{s}, t)(\frac{W}{m^2 sr})$, which is defined as energy flow per unit normal area per unit solid angle per unit time. Here, $\vec{r}$ denotes position, $\hat{s}$ denotes unit direction vector and t denotes time[22].

The RTE simply says that as a beam of radiation travels, it loses energy to absorption, gains energy by emission, and redistributes energy by scattering. In RTE, Optical properties such as refractive index n, absorption coefficient μ_a, scattering coefficient μ_s, and scattering anisotropy, g, are taken as time-invariant but may vary spatially, and Scattering is assumed to be elastic. The integro-differential form of the equation for radiative transfer is given as[23]:

$$\frac{\partial L(\vec{r}, \hat{s}, t)/\nu}{\partial t} = -\hat{s}\cdot\nabla L(\vec{r}, \hat{s}, t) - \mu_t L(\vec{r}, \hat{s}, t) + \mu_s \int_{4\pi} L(\vec{r}, \hat{s}, t) P(\hat{s}'\cdot\hat{s}) d\Omega' + S(\vec{r}, \hat{s}, t) \quad (2.2.1)$$

equation(2.2.1) describes the change of radiance, $L(\vec{r}, \hat{s}, t)$ at position $\vec{r}$ in the direction of the unit vector $\hat{s}$. Where ν is the speed of light in material medium as determined by the relative refractive index $n = \nu/c$, $\mu_t = \mu_a + \mu_s$ is total attenuation coefficient, $S(\vec{r}, \hat{s}, t)$ is a radiation source and $P(\hat{s} \cdot \hat{s}')$ is scattering phase function representing the probability of light with propagation direction $\hat{s}$ being scattered into solid angle $d\Omega$ around $\hat{s}'$. In most cases, the phase function depends only on the angle between the scattered $\hat{s}'$ and incident $\hat{s}$ directions, i.e. $P(\hat{s} \cdot \hat{s}') = P(\hat{s}' \cdot \hat{s})$. The scattering anisotropy can be expressed as g=$\int_{4\pi}(\hat{s}' \cdot \hat{s})P(\hat{s} \cdot \hat{s}')d\Omega$

The radiation transport equation above [equation(2.2.1)] can be derived by considering the radiation energy balance in arbitrary elemental volume of the medium. It is a balance equation relating the change of the energy radiation, $L(\vec{r}, \hat{s}, t)$. The left hand side term of equation (2.2.1) is the time dependent term referred to as temporal variance of radiance. The first term in the right hand side of the equation is the change of variance in the direction of the unit vector,$\hat{s}$. The expression $\mu_t L(\vec{r}, \hat{S}, t)$ represents the total attenuation of the intensity due to scattering and absorption. The integral term in right hand side of the equation is the gain due to scattering source, and the last term $S(\vec{r}, \hat{s}, t)$ is the gain due to the radiation source.

Based on the definition of radiance, there are other important physical quantities like[22];

- Fluence rate or intensity $\Phi(\vec{r}, t) = \int_{4\pi} L(\vec{r}, \hat{s}, t)d\Omega(\frac{W}{m^2})$
- Fluence $F(\vec{r}) = \int_{-\infty}^{+\infty} \Phi(\vec{r}, t)dt(\frac{J}{m^2})$
- Current density (energy flux) $\vec{J}(\vec{r}, t) = \int_{4\pi} \hat{s}L(\vec{r}, \hat{s}, t)d\Omega(\frac{W}{m^2})$. This is the vector counterpart of fluence rate pointing in the prevalent direction of energy flow.

There are no general solutions to solve the radiation transport equation due to the inherent inhomogeneities and irregularities with the physical shape of the scatterer in the medium. Even if RTE has application in wide variety of subjects including optics, astrophysics, atmospheric science and remote sensing, it's Analytic solution exist for simple cases by introducing approximations. A common approximation summarized here is the diffusion approximations. But for more realistic media with complex multiple scattering effects numerical methods are required.

2.3 Diffusion Approximations for RTE

Light propagation in a highly turbid medium, can be considered as a process of energy diffusion because the properties of light wave (polarization, interference and diffraction) are lost due to multiple scattering events.

Appropriate assumptions about the behavior of photons in a scattering medium, permits us to reduce the number of independent variables in RTE equation(2.2.1). The following assumptions lead us to the diffusion theory (and diffusion equation) for photon transport in the medium. It should be noted that the following assumptions require a high-albedo (predominantly scattering) medium[22].

a) The spectral radiance will become nearly isotropic due to Relative occurrence of absorption and scattering events. i.e relative to scattering there are very few absorption events. Likewise, after numerous scattering events, few absorption events will occur.

b) In primary scattering medium the time variation of the radiant flux density over a length of $1/\mu_s'$ (which is one transport mean free path) is assumed to be negligible with respect to the flux density itself. Thus, over one transport mean free path, the fractional change in current density is much less than unity

In diffusion theory, radiance, which can be expanded on the basis set of spherical harmonics $Y_{n,m}$, is taken to be largely isotropic, so only the isotropic and first-order anisotropic terms are used[23]:

$$L(\vec{r}, \hat{s}, t) \approx \sum_{n=0}^{1} \sum_{m=-n}^{n} L_{n,m}(\vec{r}, t) Y_{n,m}(\hat{s}) \tag{2.3.1}$$

where $L_{n,m}$ are the expansion coefficients.

Using properties of spherical harmonics and the definitions of fluence rate $\Phi(\vec{r},t)$ and current density $\vec{J}(\vec{r},t)$, the isotropic (for n=0) and anisotropic (for n=1) terms can respectively be expressed as follows:

$$L_{0,0}(\vec{r},t)Y_{0,0}(\hat{s}) = \frac{\Phi(\vec{r},t)}{4\pi} \tag{2.3.2}$$

$$\sum_{m=-1}^{1} L_{1,m}(\vec{r},t)Y_{1,m}(\hat{s}) = \frac{3}{4\pi}\vec{J}(\vec{r},t)\cdot\hat{s} \tag{2.3.3}$$

Hence we can approximate radiance by the sum of both isotropic and anisotropic terms as[23]

$$L(\vec{r},\hat{s},t) = \frac{1}{4\pi}\Phi(\vec{r},t) + \frac{3}{4\pi}\vec{J}(\vec{r},t)\cdot\hat{s} \tag{2.3.4}$$

Substituting equation (2.3.4) for radiance in RTE equation (2.2.1), integrating the scattering term of the RTE over the complete (4π) solid angle and multiplying the RTE (2.2.1) for the vector form by direction $\hat{s}$ before evaluation, the proper RTE can be respectively rewritten in scalar and vector forms as follows[22]:

$$\frac{\partial\Phi(\vec{r},t)}{\nu\partial t} + \mu_a\Phi(\vec{r},t) + \nabla\cdot\vec{J}(\vec{r},t) = S(\vec{r},t) \tag{2.3.5}$$

$$\frac{\partial\vec{J}(\vec{r},t)}{\nu\partial t} + (\mu_a+\mu_s')\vec{J}(\vec{r},t) + \frac{1}{3}\nabla\Phi(\vec{r},t) = 0 \tag{2.3.6}$$

From the second assumption of diffusion theory above, we note that the fractional change in current density $\vec{J}(\vec{r},t)$ over one transport mean free path $(1/\mu_s')$ is negligible. The vector representation of the diffusion theory RTE equation(2.3.6) reduces to Fick's law[22,23]:

$$\vec{J}(\vec{r},t) = \frac{-\nabla\Phi(\vec{r},t)}{3(\mu_a+\mu_s')} \tag{2.3.7}$$

which defines current density in terms of the gradient of fluence rate. And Substituting Fick's law equation (2.3.7) into the scalar representation of the RTE equation (2.3.5) gives the diffusion equation[22]:

$$\frac{\partial\Phi(\vec{r},t)}{c\partial t} + \mu_a\Phi(\vec{r},t) - \nabla\cdot[D\nabla\Phi(\vec{r},t)] = S(\vec{r},t) \tag{2.3.8}$$

where $D = \dfrac{1}{3(\mu_a + \mu_s')}$ is the diffusion coefficient and μ_s'=(1-g)μ_s is the reduced scattering coefficient.

2.4 Solutions of the diffusion equation with point sources in an infinite homogeneous media

For simple case, using a short-pulsed point source in an infinite homogeneous medium, the diffusion equation can be solved for fluence rate. The source term in the diffusion equation (2.3.8) above becomes $S(\vec{r}, t, \vec{r'}, t') = \delta(\vec{r} - \vec{r'})\delta(t - t')$, where $\vec{r}$ and $\vec{r'}$ are the positions at which fluence rate is measured and the position of the source respectively. and the pulse peaks at time t'.

Thus the solution can be presented as of the following[22];

$$\Phi(\vec{r}, t; \vec{r'}, t') = \frac{\nu}{[4\pi D\nu(t - t')^{3/2}]} \exp\left[-\frac{\mid \vec{r} - \vec{r'} \mid^2}{rD\nu(t - t')}\right] \exp[-\mu_a \nu(t - t')] \tag{2.4.1}$$

The exponential decay in fluence rate due to absorption in accordance with Beer's law is expressed in the equation(2.4.1) above with the term $\exp\left[-\mu_a \nu(t - t')\right]$ and the other terms represent broadening due to scattering.

Taking time variation out of the diffusion equation gives the following for the time-independent point source $S(\vec{r}) = \delta(\vec{r})$:

$$\Phi(\vec{r}) = \frac{1}{4\pi Dr} \exp(-\mu_{\text{eff}} r) \tag{2.4.2}$$

where $\mu_{\text{eff}} = \sqrt{\dfrac{\mu_a}{D}} = \sqrt{3\mu_a(\mu_a + \mu_s')}$ is the effective attenuation coefficient and indicates the rate of spatial decay in fluence.

Chapter 3

Materials and Methods

In this chapter, the materials and methods used in this work are presented. The first section of this chapter describes the various samples, chemicals and instruments used to carry out this research. In the second section of this chapter the different methodologies and methods applied to investigate optical properties and particle size of coffee samples in relation to quality and flavor are presented. The mathematical and experimental procedures used to analyze optical properties and particle sizes of coffee samples have been also reported. Some of the methodologies used here has a little similarity with the research methodologies of milk scattering experiment which is done by Senbeto[2007][24] and other researchers. even if it may have a little methodological similarity, here we used variable concentration method rather than using another external absorber like Indian ink.

3.1 Materials

Materials used in this experiment for standard sample preparation are as follows. Beakers, volumetric flasks, pipettes, measuring cylinders, magnetic stirrer with hot plate, funnel, separatory funnel, filter paper, distilled water, cylindrical glass cuvette with diameter 20.7mm and rectangular glass cuvette (10mm,10mm,30mm). Five different types of roasted grounded Arabica coffee samples in which they are at the export standard were provided by Supreme Small Holders Coffee PLC. The samples were collected from different regions of Ethiopia.Specifically $Washed\ Lekempti-2$, $Unwashed\ Harar-4$, $Washed\ Sidamo-2$, $Washed\ Yirgacheffee-2$ and $WashedLimu-2$ are used samples from their respective areas.

The laboratory apparatus and instruments used for this work are the following. 35mw He-Ne laser at 633nm wavelength, chopper, chopper controller (SR540), iris, collimator, photo diode detector, lock-in amplifier, stepper motor controller (apt) and computer interface programs for instrumental control, data recording and file handling operations.

3.2 Methods

3.2.1 Coffee sample preparation

The steps of preparing sample of coffee solution for this experimental work are as follows. All glass wares were thoroughly cleaned, rinsed with distilled water and dried before used. An accurately weighed amount of roasted grounded coffee (10g) was dissolved in distilled water and made the solution 250ml. The solution was heated and stirred for a half an hour using magnetic stirrer with hot plate. In addition the solution was filtered using a filter paper to get rid of particles from the solution. And then by weighing the filtered residue after drying, it was necessary to subtract the amount of residue from the first dissolved amount of coffee (10g) to know the amount of solute (coffee) concentration in water solvent. Here that was our concentrated stock solution. Then after that by taking a small amount of sample (3ml first, with gradually equal increase till 15ml) solution from the prepared stock solution, it was necessary to dilute each again with distilled water and made it 50ml solution to get a series of variable concentration for collimated transmission and fluence rate measurements. this was done for all five different coffee samples. The proportions of concentration are expressed using table and figure as follows:

solutions	A	B	C	D	E
Volume of stock soln. (cc)	3	6	9	12	15
Final diluted soln.(cc)	50	50	50	50	50
Concentrat ions (%)	6	12	18	24	30

Figure 3.1: *proportions of prepared solutions with gradually increased concentrations.*

3.2.2 Liquid-Liquid Caffeine Extraction

Liquid-Liquid extraction is a process of separation of components of a solution in which a solute is distributed between two immisible solvents and this process can be implemented by chloroform and dichloromethane solutions[16]. Here dichloromethane solution was used for extraction of caffeine from coffee.

The extraction process as used by Abebe[16] is used. The first step was preparing sample. an accurately weighed amount of roasted grounded coffee (300mg) was dissolved in 150ml of distilled water. The solution was stirred for a half an hour using magnetic stirrer with hot plate. In addition the solution was filtered through filter paper to get rid of particles from the solution. Then this prepared solution was mixed with dichloromethane by volume ratio(1:1). This mixture was stirred for 10 minutes, then by separatory funnel caffeine was extracted by dichloromethane from the coffee solution. The extraction was repeated for 3 times by 150ml dichloromethane at each round. Then the extracted caffeine by dichloromethane and the solution in which the caffeine was extracted from it was stored in different volumetric flasks. Then finally the attenuation coefficient, μ_t, and particle size of the caffeine extracted coffee solution were measured and compared with the normal solutions before the caffeine was extracted from it.

3.2.3 Optical Measurement techniques

The experimental work was focused on the measurement and calibration of the optical properties of the diffusing medium (coffee) such as the absorption, scattering, reduced scattering, effective attenuation coefficients and anisotropy factor. All the experiments were carried out using the continuous wave He-Ne laser (35mw at 633nm wavelength) source. In measuring the optical properties and particle size of coffee, three phases of measurements were performed. These are collimated transmission measurements, fluence rate measurements, and angular distribution of intensity measurements.

Phase-I: Collimated Transmission Measurements

Before the different concentration of coffee was examined by the system we designed, a 35mw He-Ne laser was directed to a transparent scattering cell(cuveete) of thickness 10mm containing distilled water only. The collimated transmitted light that passed through the sample was passed through the collimator. This was done to avoid the scattered light from entering the detector. in this phase the samples in which the caffeine is extracted from it and the normal solution that is coffee with caffeine is used for measurements. The experimental arrangement is shown bellow in Figure(3.2 & 3.3)

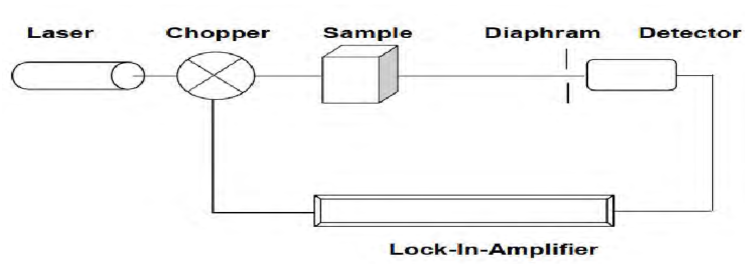


Figure 3.2: *Schematic diagram of the experimental setup for transmission measurement.*

Figure 3.3: *Photograph of the experimental set up used for transmission measurement.*

The transmitted (unscattered) light through the coffee sample was detected with photo diode detector connected to the lock-in-amplifier. The lock-in amplifier again connected to the chopper frequency controller(776Hz), which is fixed for all phase measurements, to purify the signal from other interfering frequency noise signals. The photo-diode detector used had a sensitivity curve in visible range of wavelengths. The initial intensity of light I_0, in which it was first passed though the sample cell containing only distilled water was recorded. This was done to compensate for refractive index mismatches between the external medium (air) and the surface of the cell. Then the corresponding intensities of light I, passing through different concentrations of the sample (coffee) in the light path was measured. This was done by taking a small sample solution from all diluted samples prepared first with gradually increased concentrations by 3ml(6%) until 15ml(30%) values were reached.

The experimental measurements of collimated transmission method is used to calculate the total extinction coefficient of the sample ε_t. These measurements were used to investigate the optical properties of the sample (coffee) and this would be done by applying Beer- Lamberts law.

$$I = I_0 \exp(-\mu_t L) = I_0 \exp(-c\varepsilon_t L) \tag{3.2.1}$$

Where μ_t ($\mu_t = c\varepsilon_t$) is the total attenuation coefficient, ε_t is total extinction coefficient, c is concentration of the sample and L is thickness or total path length of light through the sample in the cuvette. The relationship was plotted for $(\frac{1}{L})\ln(\frac{I_0}{I})$ against concentration(c) of the sample. From the slope of the plotted graph, the total extinction coefficient can be determined.

Measurements for the caffeine extracted coffee samples were used to determine only the attenuation coefficient (μ_t) of the sample itself. In this case we took measurement three time for average with out gradual increment of concentration and without plotting the graph. Here again similarly the thickness of the sample cuvette was 10mm. and using Beer-Laberts law equation(3.2.2) the attenuation coefficient of the given sample (Unwashed Harar-4) was calculated and presented in the next chapter.

$$I = I_0 \exp(-\mu_t L) \tag{3.2.2}$$

Phase-II: Fluence Rate Measurements

In the second phase of this experimental work a multi-angular distance measurement fluence rate or emittance, in which the rate of total amount of photons crossing over a surface of unit cross-section, inside the highly diffusing medium (coffee) was carried out. The experimental set up is shown in figure(3.4 & 3.5)

A linearly polarized He-Ne laser beam was incident on a cylindrical sample cell of diameter 20.7mm containing distilled water only to measure the initial light intensity I_0. The cylindrical sample cuvette used here was preferable because it's smooth and curved surface was necessary to get equal thickness around the sample cuveete for photo diode

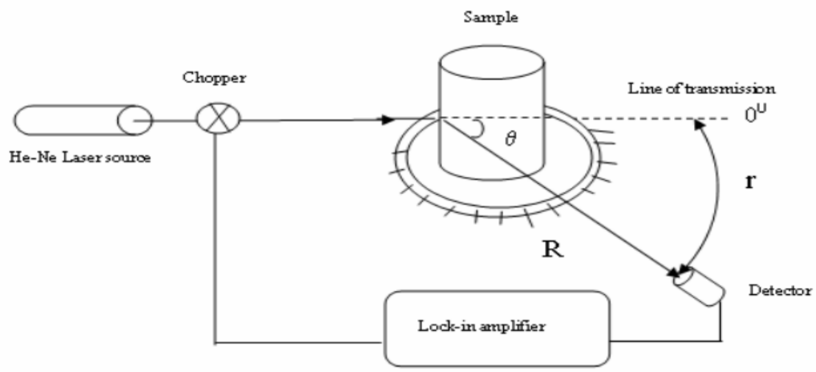


Figure 3.4: *Schematic diagram of multi-angular distance measurements set up measuring fluency rate.*

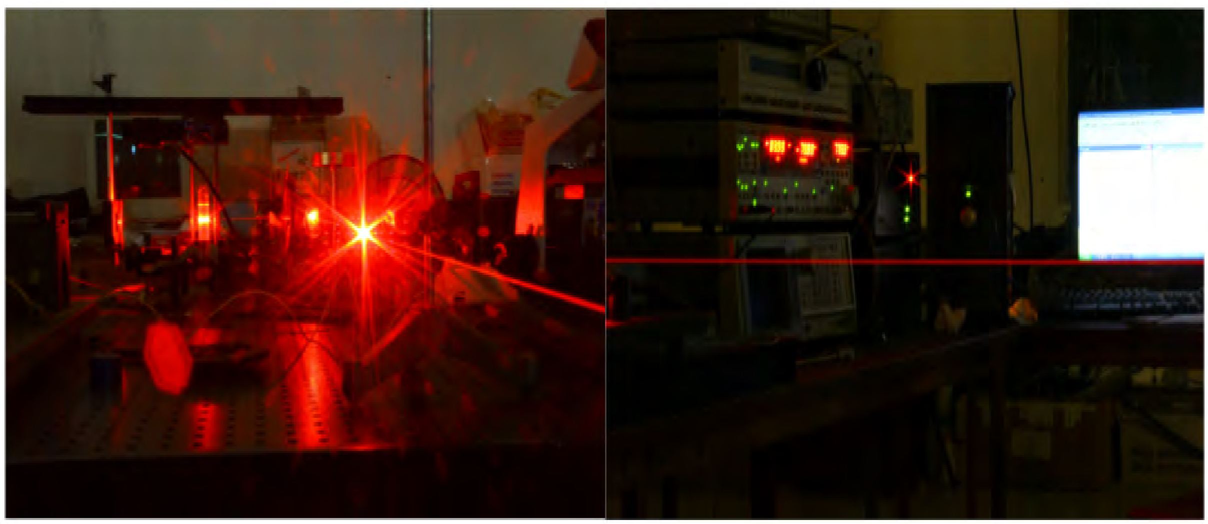


Figure 3.5: *Photograph of experimental set up for multi-angular distance measurement of fluency rate.*

detector. The corresponding intensities of laser light I, for a series of gradually increased variable concentrations were measured. The concentrations of the series of five samples used in this measurements were taken from the first prepared solutions with gradually increased concentration from 2.22% to 11.11% for different species of coffee. For each series of variably concentrated coffee samples of the added amount concentration, the fluence rate was measured by varying the angle from 27^0 to 72^0 in steps of 9^0. The angular measurement was taken from the line of transmission in both clockwise and counterclockwise directions. The source to detector distance 18cm was kept constant. The curve distance from the line of transmission to the detector at any angle θ is r= $R\theta$, which is measured using spectrometer scale. The fluence rate of the scattered light by the sample was detected by photo diode detector, and the Lock-in amplifier converted it in to amplitude signal. In this experimental phase the series of gradually increased variable concentration was used to vary the attenuation property of the medium (μ_t).

The fluence rate as a function of the curve distance between the line of transmission 0^0 and the detector (r) as it mentioned in the radiative transfer equation in chapter two is defined as

$$\phi(r) = \frac{1}{4\pi Dr} exp(-\mu_{\text{eff}} r) \tag{3.2.3}$$

Where D is the diffusion coefficient defined by $D = \frac{1}{3\mu_s'}$ for the medium having high albedo(α) and By rearranging the above fluence rate equation it can be written as follows:

$$ln(\phi(r)r) = -\mu_{\text{eff}} r + ln(\frac{3\mu_s'}{4\pi}) \tag{3.2.4}$$

For each sample of increased concentration of coffee, the graph of $ln(\phi(r)r)$ against r was plotted. And from the slope of the graph the effective attenuation coefficients μ_{eff} of the given mediums was evaluated. After the effective attenuation coefficient was found, it was possible to find the reduced scattering coefficients (μ_s') using the equation

$$\mu_{\text{eff}}^2 = 3\mu_a(\mu_a + \mu_s^{'}) \approx 3\mu_a\mu_s' \Longrightarrow for, \mu_a \ll \mu_s' \tag{3.2.5}$$

The anisotropy factor (g) can be calculated using the relations $\mu_s' = (1-g)\mu_s$ for a specific concentration. Variable concentration was used to to know the relationship between concentration and absorption or scattering coefficients of the medium. This enables us to deduce whether the medium is highly scattering or highly absorbing.

Phase-III: Angular Distribution of Intensity Measurements

In this phase we took two types of measurements as of the first phase that used for determination of particle size of coffee sample solutions with and without caffeine. These measurement has great significant to compare whether or not caffeine particles participate for scattering phenomena in the solution. Both these measurements has the same method as presented bellow. The only difference for caffeine extracted coffee sample measurements was taking sample from the stock solution itself directly without dilution with water. This was done because the concentration of the extracted caffeine can not measured directly from dichloromethane.

The first step in this phase should be measuring the incident He-Ne laser beam intensity on a sample cell containing only distilled water. A 3ml coffee stock sample solution was diluted by distilled water to make a 6% by volume 50ml solution. And then by taking 18.51ml sample from these 50ml solution the intensity measurements were carried out. The intensity of light was detected and measured three times by varying the angle around the sample from the line of transmission(0^0) clockwise and counterclockwise using the photo diode detector of the spectrometer figure(3.6 & 3.7).

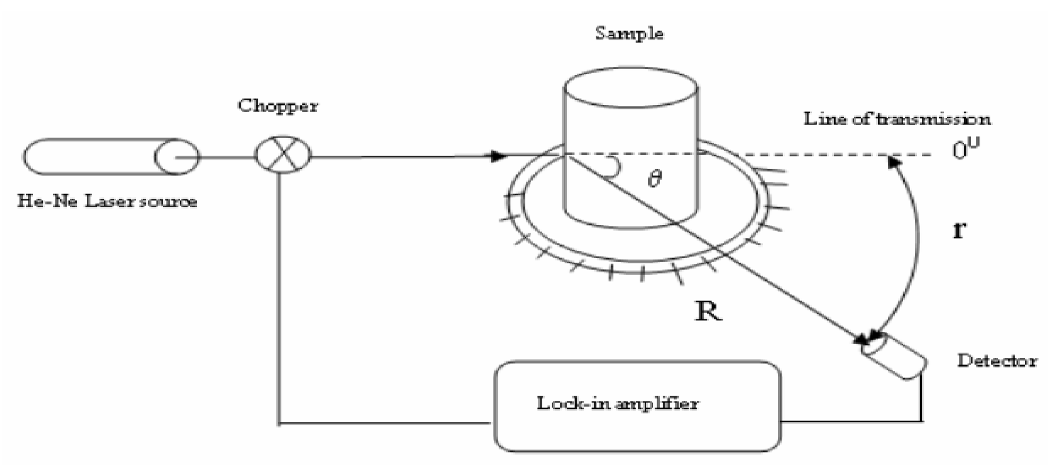


Figure 3.6: *Schematic diagram of experimental set up for angular distribution of intensity measurement.*

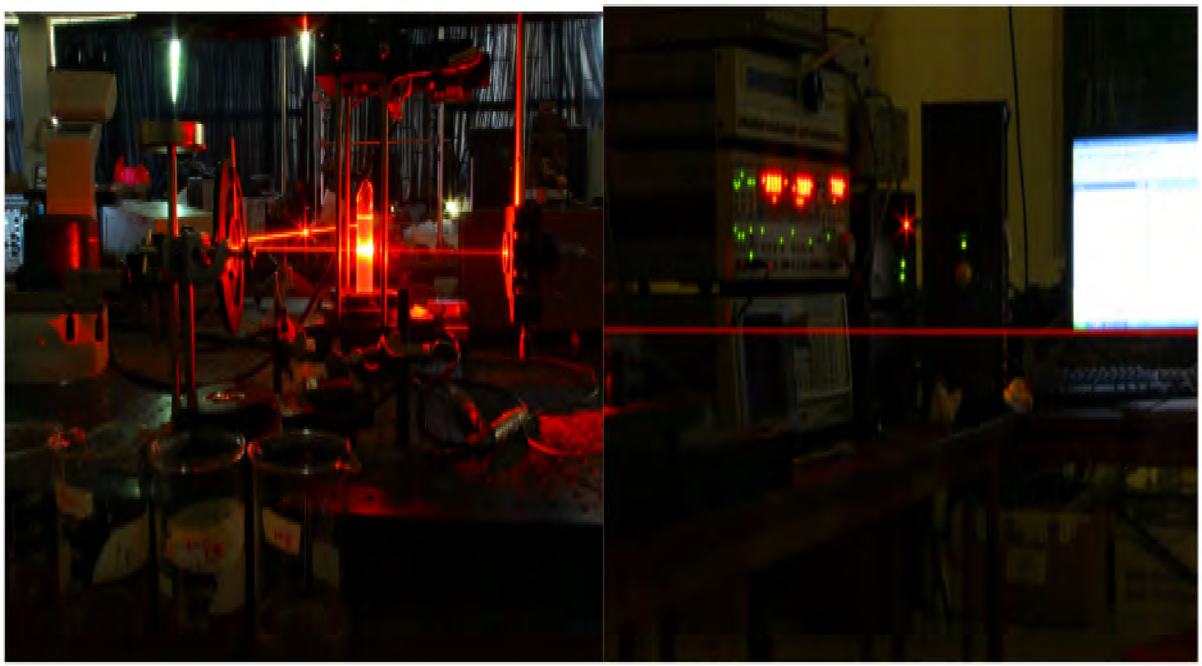


Figure 3.7: *Photograph of experimental set up for angular distribution of intensity measurement.*

The Airy disk (or a diffuse circular disk) would be formed from the distribution of detected light intensity rather than forming a bright dot as image when light from He-Ne laser source passes through spherical scattering particle[15]. The diameter of the Airys disc corresponds to the diameter of the scattering particle. the size of the particle can be calculated from the angle measurement and using the diffraction equation

$$d = \frac{1.22\lambda_0}{\theta} \tag{3.2.6}$$

The spherical scattering particle size(diameter) can be computed using the value of d. θ in the above expression represent the angle from the transmission line 0^0 to where the first minima occur. The particle size parameter can then be calculated as

$$\chi = \frac{d\pi}{\lambda_0} n_{med} \tag{3.2.7}$$

where n_{med} is the refractive index of the surrounding medium (water≈1.33 at room temperature) and λ_0 is the wavelength of the incident He-Ne laser beam.

A Mie scattering theory of particle size calculation again yields the scattering efficiency Q_s of the scatterer in the solution to know the amount of efficient cross-sectional area for scattering phenomena as follows

$$Q_s = \frac{\mu_s}{\rho_s A} \tag{3.2.8}$$

where A is the true geometrical area of the scatterer and ρ_s is the particle number density which relates the scattering cross sectional area σ_s to the true geometrical area ($A = 4\pi r^2$) of the spherical particle by the equation

$$\sigma_s = \frac{\mu_s}{\rho_s} = Q_s A \tag{3.2.9}$$

After the particle sizes of different species of coffee samples were determined from the experiment, it was tried to relate each with their quality and flavor standards given by quality and standard inspection centers. This give us information about the particle size dependency of quality and flavor of coffee. And this will be discussed briefly in the discussion part of chapter four next.

Chapter 4

Results and Discussions

In this chapter the results found from the three phases of experiments are presented independently. The analysis was performed by Beer-Lamberts Law and RTE in the diffusion approximation theory. Graphs and some algebraic calculations are used in the discussion to show the relationship between various quantities. The relationships between concentration & absorbance was used to calculate the extinction coefficient of the given coffee sample medium. Curve distance & fluency rate graph is used to determine scattering, reduced scattering and absorption coefficients and anisotropy factor. The graph of angular distance, θ, versus intensity with diffraction equation was used to calculate the particle size and scattering parameter of the sample.

Finally comparisons of particle sizes of the caffeine extracted coffee sample with the sample solution without caffeine is extracted would be performed. And also the calculated particle size of the given sample is compared with the grade of coffee given by traditional cup test method.

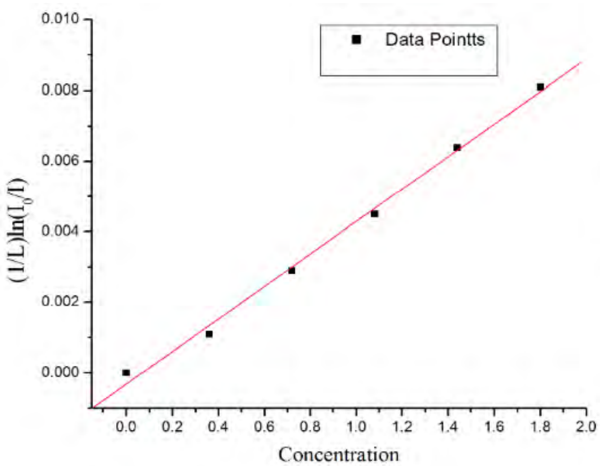
4.1 Total Extinction Coefficients

The collimated transmission measurement phase of the experiment, section (3.2.3) phase-I is used to calculate the total extinction coefficients of the prepared sample coffee medium of different species. In all five diluted samples, the concentration of coffee was varied from 6% to 30% with 6% variation by volume. From each of all these, a 3ml series of samples having a concentration increment of 0.36% (from 0.36% to 1.8%) were taken by a rectangular cuvette for the measurement. The initial incident intensity I_0 was measured with the sample cuvette containing only the solvent, which was distilled water and found to be $I_0 = 117.92$a.u. Then the corresponding intensities of light I, passing through different concentrations of the sample (coffee) in the light path was measured. The experimental results for all five coffee species with different concentrations of prepared stock sample solutions in which $Unwashed\ Harar - 4$(10mg/ml), $Washed\ Lekempti -$ $2(10.2mg/ml)$, $Washed\ Sidamo - 2(10.4mg/ml)$, $Washed\ Yirgacheffee - 2$(10.4mg/ml) and $WashedLimu - 2(11mg/ml)$ were shown in Table 4.4 in Appendix A.

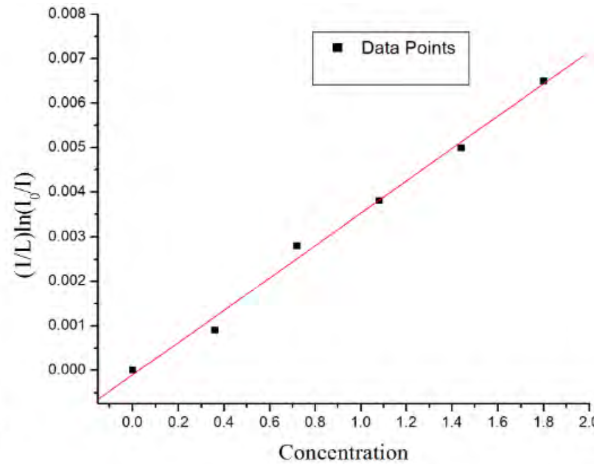
Using Beer-Lamberts law equation(3.2.1), the relationship was plotted for $(1/L)ln(I_0/I)$ against concentration, figure(4.1). The linearity of the results shows that Beer-Lamberts law is well applicable in the range of the concentrations studied with 0.9976 regression coefficient and ±0.001 error. The slope of the straight line represents the total extinction coefficient ε_t , which is the attenuation of the intensity of light due to absorption and scattering in the sample. The total extinction coefficients ε_t for different samples are given in Table (4.1).

Types of Coffee Samples	Total Extinction Coefficients ε_t(mm^{-1})
Unwashed Harar-4	4.651x10^{-3}
Washed Lekempti-2	3.635x10^{-3}
Washed Sidamo-2	7.823x10^{-3}
Washed Yirgacheffee-2	8.582x10^{-3}
Washed Limu-2	7.042x10^{-3}

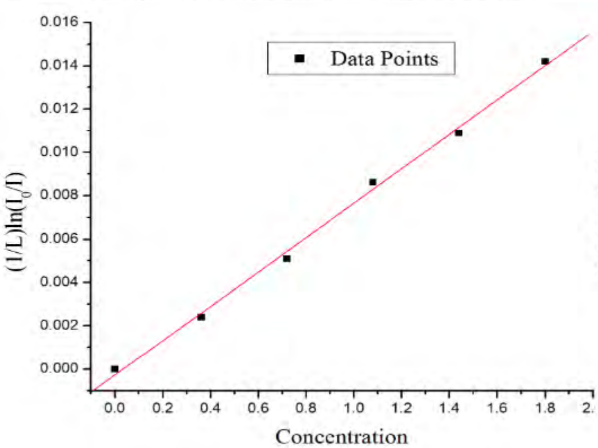
Table 4.1: *Results of total extinction coefficients of different species of coffee samples*



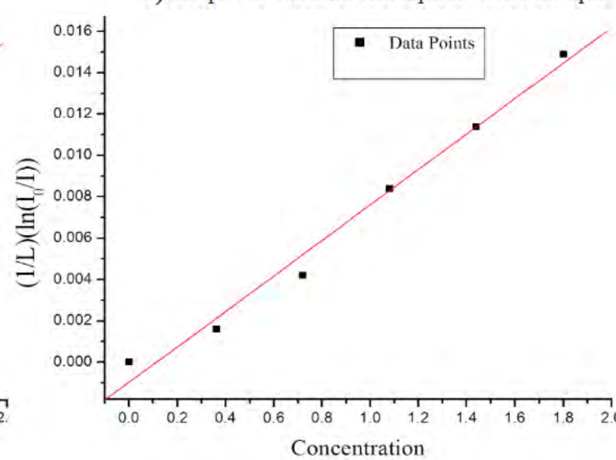
a) Graph For Unwashed Harar-4 Coffee Sample



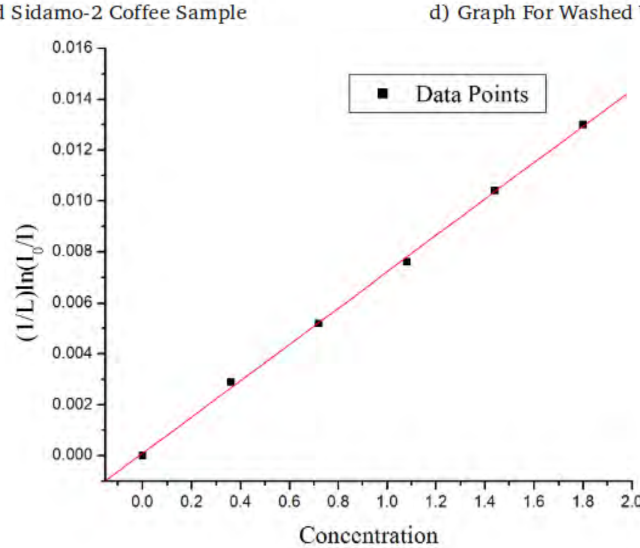
b) Graph For Washed Lekempti-2 Coffee Sample



C) Graph For Washed Sidamo-2 Coffee Sample



d) Graph For Washed Yirgacheffee-2 Coffee Sample



e) Graph For Washed Limu-2 Coffee Sample

Figure 4.1: *graphs of $(1/L)ln(I_0/I)$ versus concentration C of five different species of coffee samples used in this experiment*

As it can be observed from Table(4.1) it is necessary to generalize as different species of coffee grown in Ethiopia has different attenuation coefficient in the given wavelength of light. This generalization intern leads us to deduce that the quality and flavor of different species of coffee can be affected by this property with relation to absorption and scattering coefficients. As it can be seen from the table washed Yirgacheffee-2 and washed lekempti-2 coffee has a highest and lowest extinction coefficient respectively.

The total attenuation coefficient μ_t of caffeine extracted coffee sample were calculated using equation(3.2.2). In this step of experiment only Unwashed Harar-4 coffee type was used with 10mm path length of light (or thickness) in the rectangular cuvette. The corresponding intensity for the given sample was detected as $I = 108.61$a.u. And the result of calculation from the experiment gives us the total attenuation coefficient μ_t of Unwashed Harar-4 coffee sample was 8.22x10^{-3}mm^{-1}.

4.2 Absorption, Scattering and Reduced Scattering Coefficients And Anisotropy Factors

Multi angular distance or fluence rate measurement method was carried out to investigate absorption, scattering and reduced scattering coefficients and anisotropy factors of used coffee sample mediums. This phase measurement results were analyzed based on the solution of the radiation transport equation (RTE) for a point source in an infinite medium with diffusion equation (DE) approximations. The concentrations of coffee were varied from 2.22% to 11.11% in volume ratio for which the fluence rate intensity $\phi(r)$ was attenuated. The raw data of the measurements were presented in Table(4.5) in Appendix B, and their corresponding graphs for $ln(\phi(r)r)$ versus r are shown as figure(4.2).

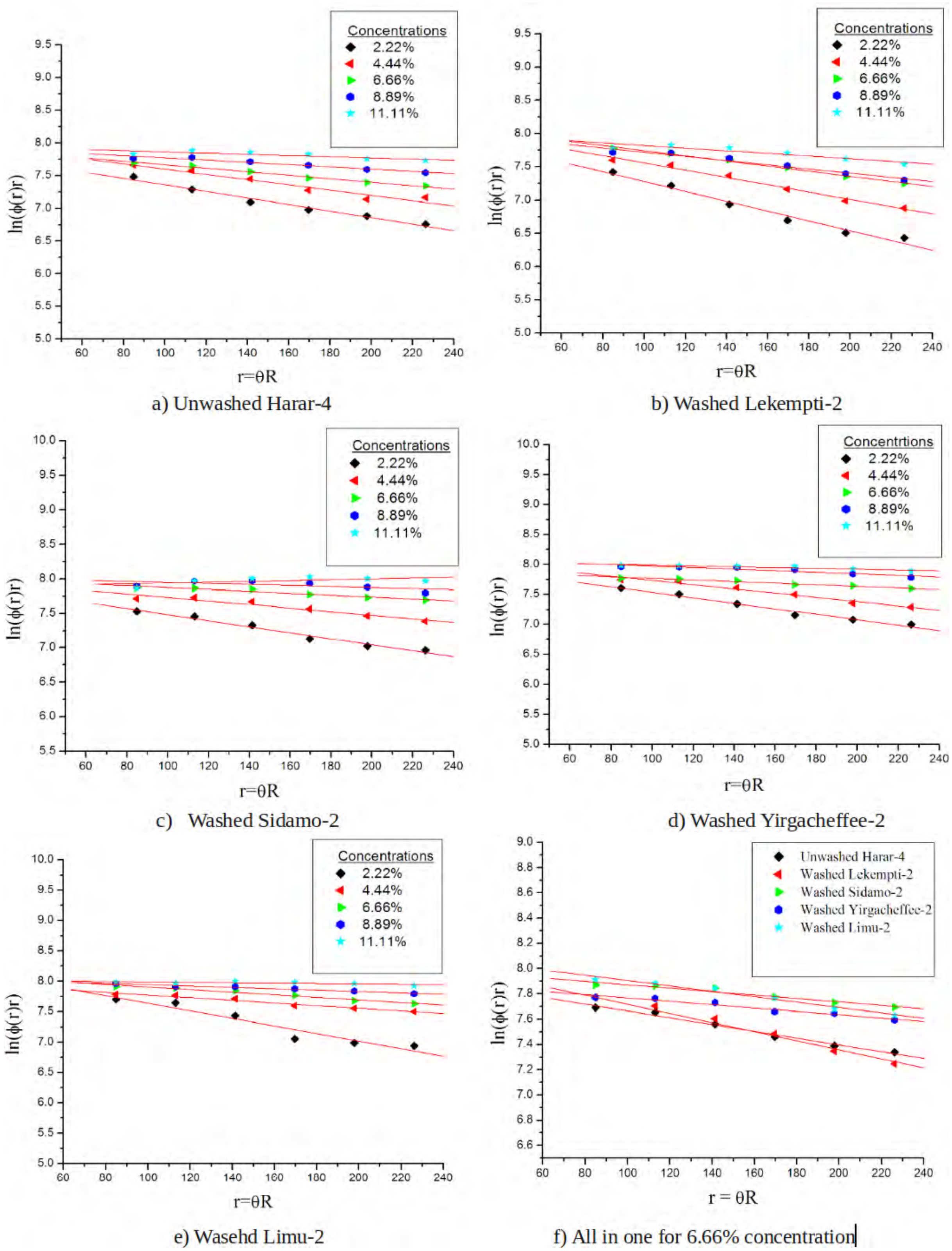


Figure 4.2: *graphs for fluence rate measurements($ln(\phi(r)r)$ versus r) for all five different types of coffee samples used in the experiment*

From the raw data values in the table and from each curve of graphs a, b, c, d and e in figure(4.2), it can be observed that the fluence rate increases as the concentration of the medium(coffee) increases. The lower line from each graph(a,b,c,d, and e) signify measurements made with lower concentration of coffee sample medium in water solution. The slopes of these curves in the graph are increasing according to the increase in concentration for each types of coffee samples. This indicates that all five samples of coffee used in this experiment have high albedo constant or it has no absorption coefficient($\mu_a = 0$) for this given wavelength value of light, in which 633nm with 35mw He-Ne laser. This shows that the results are in good agreement with the diffusion equation approximations in the literature in which high albedo medium is necessary to use those approximations.

To calculate the effective attenuation coefficient(μ_{eff}) and reduced scattering coefficient ($\mu_s^{'}$) from the slope of each graphs and equation(3.2.5) respectively, a curve with specific concentration of the scatterer 6.66% by volume is selected from each samples of coffee graphs(a,b,c,d and e) and plotted as graph (f) together in figure(4.2). The evaluation of the slope of $ln(\phi(r)r)$ as a function of distance, r, gives us the effective attenuation coefficient $\mu_{\text{eff}}^2 = 3\mu_a\mu_s^{'}$ which intern is used to find the reduced scattering coefficient.

Referring $\mu_{\text{eff}}^2 = 3\mu_a\mu_s^{'}$ in particular, the $\mu_s^{'}$ is simply proportional to the concentration of scatterers, where as the absorption coefficient in the scattering medium is due to only the absorption of the distilled water used for dilution which has an absorption coefficient of 2.6x10^{-4} at 633nm wavelength[25,26].

Since $\mu_a = \mu_{as} + \mu_{aw}$ and $\mu_{as} = 0$, where μ_{as} and μ_{aw} are the absorption due to both scatterer and pure water respectively, the the reduced scattering coefficient $\mu_s^{'}$ of all coffee samples are presented in table(4.2) by using $\mu_s^{'} = \mu_{\text{eff}}^2/3\mu_{aw}$.

Assuming the loss in intensity due to reflection is minimum, the total extinction coefficient is as a result of absorption and scattering. And since the absorption coefficient of scatterer is zero ($\mu_a = 0$), the total extinction coefficient is equal to the extinction coefficient due to scattering only ($\varepsilon_t = \varepsilon_s$). Hence multiplying ε_s, which is obtained using phase-I measurement results of the experiment, with concentration c results in the scattering coefficient μ_s of the scatterer. Using this value and the value obtained for the reduced scattering coefficient, the magnitude of the anisotropy factor g for each types of coffee was computed. All the results of necessary parameter calculations are summarized in Table(4.2) as follows:

Types of Coffee	C (%)	$\mu_{\text{eff}}(\text{mm}^{-1})$	$\mu_s'(\text{mm}^{-1})$	$\mu_s(\text{mm}^{-1})$	g-value
Unwashed Harar-4	6.66	2.672x10^{-3}	0.915x10^{-2}	3.098x10^{-2}	0.999
Washed Lekempti-2	6.66	3.383x10^{-3}	1.467x10^{-2}	2.421x10^{-2}	0.999
Washed Sidamo-2	6.66	1.396x10^{-3}	0.250x10^{-2}	5.210x10^{-2}	0.999
Washed Yirgacheffee-2	6.66	1.383x10^{-3}	0.245x10^{-2}	5.716x10^{-2}	0.999
Washed Limu-2	6.66	2.240x10^{-3}	0.643x10^{-2}	4.690x10^{-2}	0.999

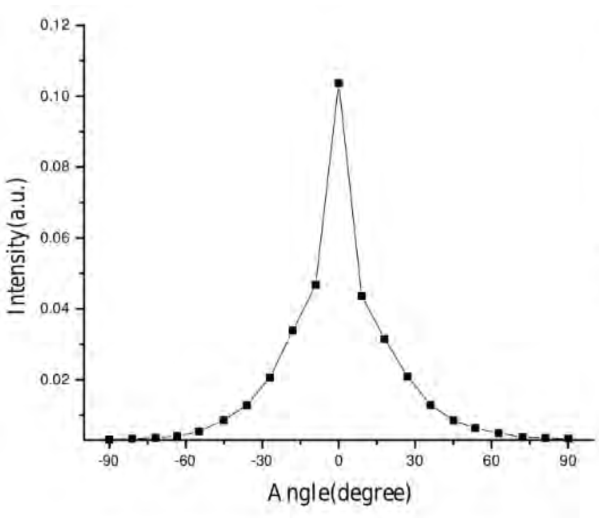
Table 4.2: *Summery for calculation values of different optical properties of coffee samples used in the experiment*

Table (4.2) shows that all coffee types with different quality and flavor have different values for the different optical quantities, i.e. extinction coefficient, scattering coefficient, reduced scattering coefficient and anisotropy factor. Accordingly, Washed Yirgacheffee-2 coffee has highest value of scattering coefficient than all other four coffee species, and that of Washed Lekempti-2 has the least value of scattering coefficient. The result revealed that the anisotropy factor values for all coffee types are almost similar in which all scattering phenomenon were in the forward direction. All calculations have been done in this phase precisely with averages of ± 0.0015 error and 0.98 regression coefficient for all graphs.

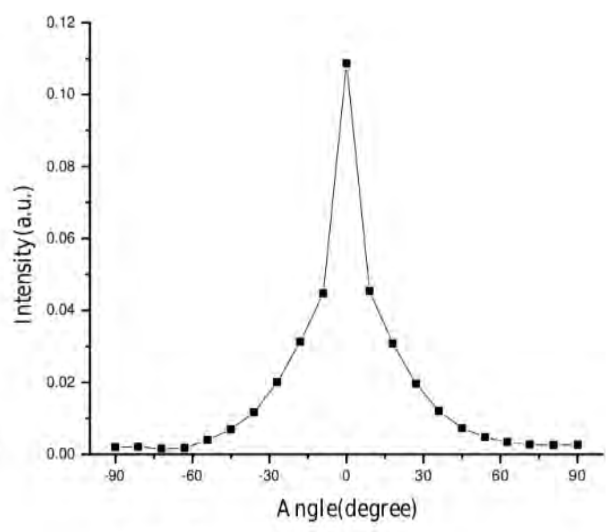
4.3 Particle Size Measurement Results

Determination of diameter (or particle size) of the scattering particle and particle size parameter χ can be performed using the results of the 3^{rd} phase measurements of the experiment, angular distribution of intensity measurements. Each five types of coffee stock samples used in this phase experiment were diluted to have a concentration of 6% by volume in a 50ml solution. And then an 18.51ml (concentration by volume) of sample was taken by cylindrical cuvette from these diluted 50ml solution to get a volume fraction of $f_v = 0.0222$. And this is used to calculate the particle number density ρ_s of the scatterer in the sample solution when it is divided by the true geometrical volume of the scatterer. The particle number density ρ_s intern is used to calculate the scattering efficiency Q_s and the scattering cross-sectional area σ_s using equations(3.2.9 & and 3.2.10).

In this experiment, the intensity distribution is symmetric and centered at 0^0 and the airy pattern and airy disk is formed around this center. Figure (4.3) shows the results of symmetric distribution of scattered light intensity around a small suspended particle in the solution. By taking the angle from the graphs where the first minima occurs and using equations(3.2.7 & 3.2.8), the particle size (or diameter 'd' of the particle), particle size parameter χ and other necessary parameters were calculated precisely and the results were presented in table (4.3).



a) Graph For Unwashed Harar-4 coffee Sample



b) Graph For Washed Lekempti-2 Coffee Sample

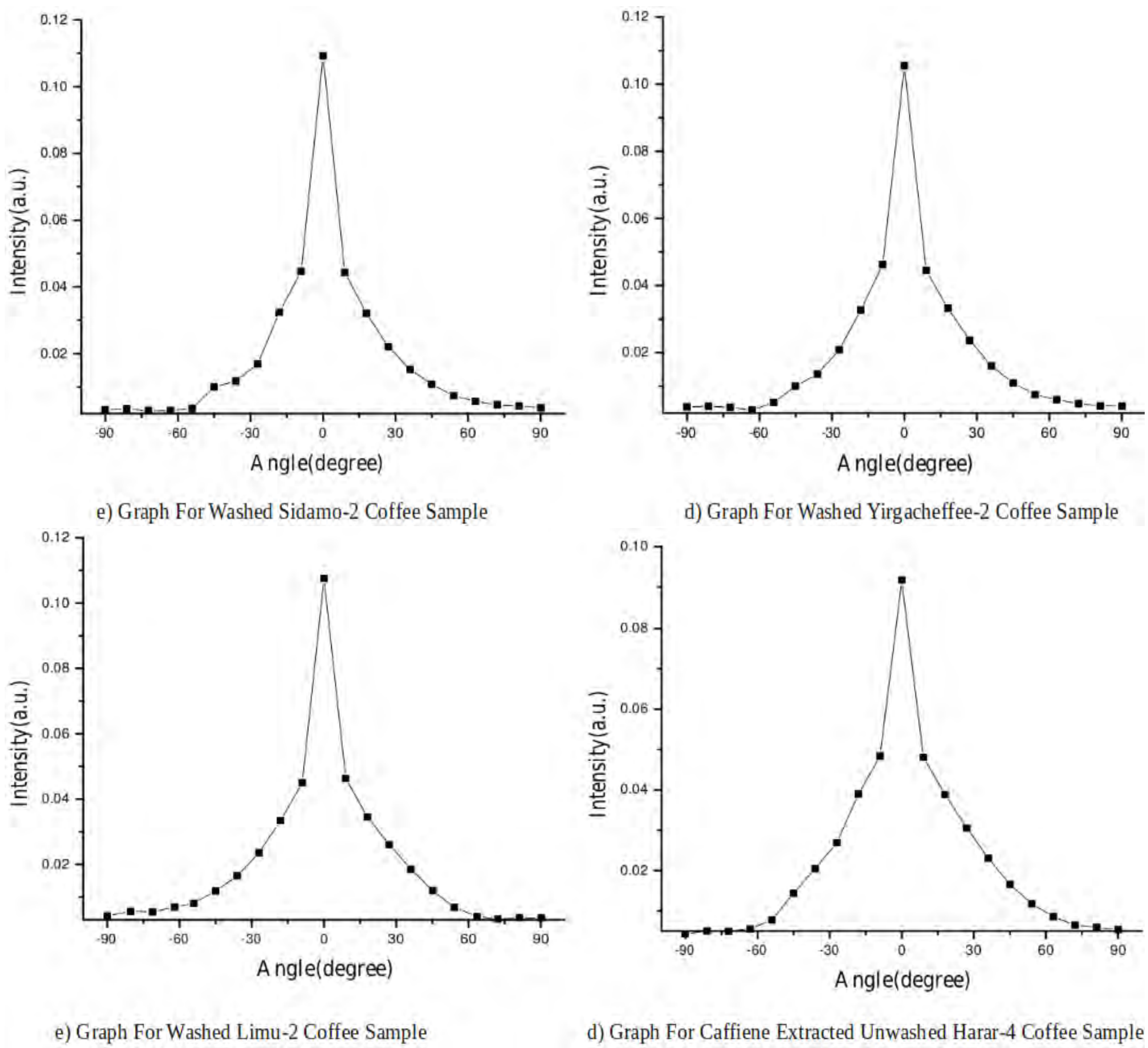


Figure 4.3: *Graphs of symmetric angular distribution of intensity measurements of five different coffee samples used in the experiment*

Types of Coffee Samples	$\theta(^0)$	d(μm)	χ	A(μm^2)	$\rho_s(\mu m^{-3})$	Q_s	$\sigma_s(\mu m^2)$
Unwashed Harar-4	42.59	1.039	5.157	3.391	3.780×10^{-2}	2.417×10^{-4}	8.196×10^{-4}
Wasehd Lekempti-2	40.77	1.085	5.385	3.698	3.319×10^{-2}	2.151×10^{-4}	7.294×10^{-4}
Wasehd Sidamo-2	47.98	0.922	4.576	2.671	5.410×10^{-2}	3.606×10^{-4}	9.630×10^{-4}
Washed Yirgacheffee-2	59.17	0.900	4.467	2.545	5.816×10^{-2}	3.862×10^{-4}	9.828×10^{-4}
Washed Limu-2	48.57	0.911	4.521	2.607	5.608×10^{-2}	3.208×10^{-4}	8.363×10^{-4}

Table 4.3: *calculation summery of different parameters related to scattering of light by different coffee type samples used in the experiment*

Particle sizes for different coffee species are different according to the results obtained in the experiment. It can be observed from the table Washed Yirgacheffee-2 coffee has the most finest particle in it and that of Washed Lekempti-2 has the largest particle size from all others. The results obtained in phase-II measurements also revealed that coffee sample with finest particle size has highest scattering coefficient. According to rate of chemical reaction theory in chemistry the finest particle has fastest rate in its reaction. This shows that Washed Yirgacheffee-2 coffee has fastest rate to release good flavor than others. And this generalization is in good agreement with that of traditional cup-test method grading system in which the grading as supplied by Supreme Small Holders Coffee PLC, Washed Yirgacheffee-2 (1^{st}), Washed LImu-2 (2^{nd}), Washed Sidamo-2 (3^{rd}), Unwashed Harar-4 (4^{th}) and Washed Lekempti-2 (5^{th}). Thus, the results determined from the experiment has order of grades as they are listed in table(4.3) with their corresponding particle size (diameter), particle size parameter and other related parameters. This indicates that the finest the particle release the best is the flavor.

The particle size of Caffeine extracted Unwashed Harar-4 coffee is measured and calculated in similar procedure. The scattering angle obtained form the measurement is 59.95^0 and the result of calculation gives d=0.738μm, and it's particle size parameter $\chi = 3.663$ with geometrical area 1.711μm^2. From these result of calculation it can be conclude that caffeine in the coffee has its own effect on the scattering of light, and also it affects the quality and flavor of coffee.

In general in this series of measurements it may be mentioned that there is an average error of $\pm 0.001\mu$m in particle size determinations. Results of particle size parameter χ, tells us that our results agreed with Mie scattering theory in which they all have $\chi > 1$ or particle size is greater than the wavelength of the incident light which is 633nm He-Ne laser for our experiment.

Chapter 5

Conclusion and Future Outlooks

The method for investigation of Particle size dependency of quality and flavor of coffee and it's optical properties, (ε_t, μ_a, μ_s, μ_s' and g), have been developed in this experimental research. Light scattering technique without external absorber method using a 35mw He-Ne at 633nm wavelength was carried out for investigation.

The results for all used coffee samples confirm that the diffusing medium, coffee, has a high and dominant scattering property in the forward direction with negligible absorption property for the given wavelength of incident light. The particle size results of different coffee samples showed that finest particle size related to good quality and flavor with highest scattering coefficient as per the information supplied by Supreme Small Holders Coffee PLC using traditional cup-test method. Accordingly washed Yirgacheffee-2 is the first in quality with succession of others. And the results also revealed that caffeine has its own effect on scattering.

The developed method for investigation is simple, fast, cheap and valid. More over it is highly sensitive and more suitable for on line monitoring with equipments mostly available in common laboratories. Since the developed method have wider economic and scientific applications, even for other turbid mediums, light scattering method is the most preferable for classification of coffee quality standards. Further research may improve it, if necessary, for other mediums. Thus it is recommended for quality inspection centers and coffee exporters to use light scattering techniques for their quality standard classifications.

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Appendices

Appendix A: Raw Data For Collimated Transmission Intensity Measurements

Stock soln.⇨ / Diluted Sample soln. ⇩	Unwashed Harar-4 (10mg/ml)		Washed Lekempti-2 (10.2mg/ml)		Washed Sidamo-2 (10.4mg/ml)		Washed Yirgacheffee-2 (10.4mg/ml)		Washed Limu-2 (11mg/ml)	
Concentration(%)	Intensity(a.u.)	$(1/L)\ln(I_0/I)$	Intensity(a.u.)	$(1/L)\ln(I_0/I)$	Intensity(a.u.)	$(1/L)\ln(I_0/I)$	Intensity(a.u.)	$(1/L)\ln(I_0/I)$	Intensity(a.u.)	$(1/L)\ln(I_0/I)$
0	117.92	0.00	117.92	0.00	117.92	0.00	117.92	0.00	117.92	0.00
0.36	116.61	0.0011	116.86	0.0009	115.14	0.0024	116.01	0.0016	114.55	0.0029
0.72	114.50	0.0029	114.68	0.0028	112.06	0.0051	113.11	0.0042	111.93	0.0052
1.08	112.73	0.0045	113.52	0.0038	108.20	0.0086	108.39	0.0084	109.29	0.0076
1.44	110.61	0.0064	112.17	0.0050	105.74	0.0109	105.23	0.0114	106.27	0.0104
1.80	108.70	0.0081	110.50	0.0065	102.35	0.0142	101.62	0.0149	103.55	0.0130

Table 4.4: *Transmission intensity measurements for different concentrations of coffee type samples*

Appendix B: Raw Data For Fluence Rate Measurements

Types of coffee	Angle θ^0	Curve Distance $r=R\theta$(mm)	$\phi(r)$ $\Delta C=2.22\%$	$\phi(r)$ $\Delta C=4.44\%$	$\phi(r)$ $\Delta C=6.66\%$	$\phi(r)$ $\Delta C=8.89\%$	$\phi(r)$ $\Delta C=11.11\%$
Unwashed Harar-4	27	84.82	20.91	25.02	25.82	27.83	29.57
	36	113.09	12.91	17.21	18.65	21.03	23.35
	45	141.37	8.51	12.12	13.58	15.78	18.19
	54	169.64	6.32	8.48	10.25	12.48	14.68
	63	197.91	4.94	6.35	8.18	10.04	11.78
	72	226.19	3.81	5.71	6.81	8.36	10.04
Washed Lekempti-2	27	84.82	19.65	23.53	27.98	26.46	28.38
	36	113.09	12.02	16.39	19.59	19.59	22.1
	45	141.37	7.29	11.17	14.22	14.53	17.03
	54	169.64	4.76	7.60	10.5	10.80	13.09
	63	197.91	3.39	5.49	7.84	8.24	10.25
	72	226.19	2.75	4.3	6.20	6.53	8.27
Washed Sidamo-2	27	84.82	21.94	26.40	30.84	31.52	30.82
	36	113.09	15.29	20.08	22.95	25.42	25.57
	45	141.37	10.77	15.14	18.07	20.51	21.24
	54	169.64	7.36	11.32	14.01	16.48	18.07
	63	197.91	5.65	8.79	11.51	13.28	15.14
	72	226.19	4.67	7.14	9.71	10.74	12.73
Washed Yirgacheffee-2	27	84.82	23.68	27.23	27.95	33.85	34.68
	36	113.09	16.02	19.99	20.78	25.21	25.77
	45	141.37	10.96	14.34	16.08	20.08	20.40
	54	169.64	7.54	10.62	12.51	16.14	16.97
	63	197.91	5.98	7.90	10.56	12.85	13.95
	72	226.19	4.82	6.47	8.79	10.62	11.78
Washed Limu-2	27	84.82	26.06	28.20	32.21	33.88	34.49
	36	113.09	18.49	20.87	23.50	23.99	25.48
	45	141.37	11.96	15.84	18.04	19.2	21.06
	54	169.64	6.84	11.75	13.92	15.47	17.27
	63	197.91	5.48	9.64	10.93	12.79	14.47
	72	226.19	4.57	8.00	9.120	10.71	12.24

Table 4.5: *fluence rate measurement raw data of all five types of coffee samples used in the experiment*

Declaration

I, the undersigned, declare that this research is my original work, has not been presented for a degree in any other University and that all the sources of material used for the research have been dully acknowledged.

Name: Endris Taju

Signature: ______________________

Place and time of submission: Addis Ababa University , June 2012

This research has been submitted for examination with my approval as University advisor:

Name: Prof. A. V. Gholap

Signature: ______________________