Fabrication and Experimental Analysis of Mechanical Properties of Aluminium (AA6063) Alloy/Eggshell Composite

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Name of co-Advisor Advisor	Signature	Date

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ABSTRACT

The main purpose of this study is to fabricate the metal matrix composite using carbonized eggshell powders as reinforcement and AA0663 alloy as matrix material to study the mechanical properties of composite specimens. To achieve the above purpose, stir casting method was used for development and processing of AA6063 alloy/eggshell composite. It is aimed to provide optimum stir casting process of composite thereby reduce product cost and improve mechanical properties, particularly in B and C Aluminum Company. The Taguchi method is used to design of experiment and optimize the process. Each parameter has three different levels. Taguchi method orthogonal array of L27 (3^4) is used to design this experiments. The experimental investigation was conducted under varying input influential process parameters while taking constant other stir casting parameters for resulted tensile strength and hardness test were done. The study reveals that the optimum setting values of eggshell weight percentage, stirring speed, stirring time and stirring temperature were determined as, 10 wt.%, 200 rpm, 10 minutes and 750 ^{o}C, respectively. The level of significance of each parameter was determined by the help of analysis of variance, so that eggshell weight percentage contributes 50.12 %, stirring speed 10.14 % stirring time 27.44 % and stirring temperature 8.87 % for tensile strength while eggshell weight percentage contributes 68.82 %, stirring speed 7.51 % stirring time 15.69 % and stirring temperature 3.88 % for hardness respectively.

The naturally occurring eggshell material is carbonized and added to aluminum (AA6063) alloy by stir casting technique and its effects on the microstructures and mechanical properties of the composite were studied. The microstructure of the carbonized eggshell/AA6063 alloy composite was examined with the aids of optical microscopy after the specimen surface has been carefully prepared. Optical micrographs revealed that there was a homogenous distribution of eggshell particles within the aluminum (AA6063) alloy. The density of the composite also reduced significantly with an increase in the composition of the carbonized eggshell in the aluminum (AA6063) alloy. The eggshell powders can be used as reinforcement in aluminum (AA6063) alloy and its very less expensive reinforcement material. The results revealed that by the addition of carbonized eggshell particles up to 10 wt.% in AA6063 alloy, the tensile strength and hardness were increased. It was also noted that the tensile strength and hardness of the composite has decreased with the addition of the carbonized eggshell particles above 10 wt.% in the aluminum (AA6063) alloy. These results showed that the mechanical properties (tensile strength and hardness) of aluminum (AA6063) alloy are improved after the addition of carbonized eggshell powders.

Keywords: Aluminum - alloy, eggshell; composite; stir casting; carbonized eggshell; mechanical properties; microstructure.

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ABBREVIATIONS AND ACRONYMS

AA Aluminum Association

AMCs Aluminum Metal composites

AMMC Aluminum Metal Matrix Composite

ANOVA Analysis of Variance

BHN Brinell Hardness Number

CES Carbonized Eggshell

DOE Design of Experiment

DOF Degree of Freedom

ES Eggshell

ESP Eggshell powder

HRB Rockwell Hardness Number

MMCs Metal Matrix Composites

MSE Mean Square Error

OM Optical Microscope

ROM Rule of Mixture

RSH Rice Husk Ash

S/N Signal to Noise

SST Total Sum of Square

SSTR Sum of Square or Treatment Sum of Square

UTM Universal Tensile Machine

UTS Ultimate Tensile Strength

VHN Vickers Hardness Number

YS Yield Strength

CHAPTER 1: INTRODUCTION

1.1. Background of the Study

The modern development in the field of science and technology demands the developments of advanced engineering materials for various engineering applications, especially in the field of transportation, aerospace, architectural, building products, door and window frames, pipe and tube for irrigation systems, ladders and military engineering related areas. The need of materials with optimum properties and cost for various engineering applications has paced research in area of metal matrix composites for the past many decades. These area demand light weight-high strength materials [1]. Such demands can be met by development and processing of aluminum metal matrix composite materials. Aluminum, both in pure and alloy form is a suitable material for many engineering and industrial applications. Thus, the physical, chemical and mechanical properties of aluminum have fascinated researchers to explore its potential. In aluminum matrix composites, the extensively used reinforcement is in particulate form and is widely employed in automotive and aerospace industries [2].

Composite materials have been utilized to solve technological problems for a long time. In 1960s these materials start capturing the attention of industries with the introduction of polymeric-based composites. Since then, composite materials have become common engineering materials and are designed and manufactured for various applications. The growth in composite usage also came about of increased awareness regarding product performance and increased competition in the global market for lightweight components [3]. Among all materials, composite materials have the potential to replace widely used steel and aluminum (aluminum alloys) with better performance. Replacing steel components with composite components can save 60 to 80% in component weight and 20 to 50% weight by replacing aluminum parts [4]. Today, it appears that composites are the materials of choice for many engineering applications.

The properties of a composite depend on the followings;

- Properties of constituent phases.
- Relative amounts of constituents.
- Geometry of the dispersed phase (Shape of particles, Particle size, Particle distribution).

Several properties of the composites may be assessed by the rule of mixture (ROM), which states that the property of the composite is the sum of products of the property of an individual constituent and its volume fraction in the composite. Metal matrix composites (MMCs) exhibit the ability to withstand high tensile and compressive stresses by the transfer and distribution of the applied load from the ductile matrix to the reinforcement phase. A material is called composite if it has two or more distinct phases and the properties vary largely from the individual phase. Composites contain two phases, matrix phase and dispersed phase [5].

The primary phase is called matrix phase, this phase is continuous and it is more ductile and less hard it holds the secondary phase and shares the load with it. The secondary phase is discontinuous form and is called dispersed phase. Usually this phase is harder than primary so it is also called reinforced phase. Generally Composite materials can be defined that are formed by the combination more materials to achieve properties that are superior to those of their constituents [6].

In present days, automobile industries are rapidly increased. All the automobile industries are in the process of supplying the vehicles at low cost. The major cost of the automobile components is its body parts. As per the European Union and Asian countries automotive components end life is very important factor in the design of the automotive [7]. So everybody is concentrating on materials which will be strong enough, less weight, recyclable with reduced cost. Hence all the researchers are concentrated on the composite materials which have all the above properties. Generally, the composite materials are synthetic and natural. In that natural fiber is ahead of synthetic with their low cost, low density, stiffness, fairly mechanical properties, high specific strength, non-abrasive, ecofriendly and biodegradable characteristics.

The aluminum metal matrix composite materials are the combination of two or more constituents in which aluminum metal is matrix and other is filler materials (reinforcements). Advances on lightweight and high performance structural components are important enablers for light weighting and in the transportation sector [8]. Aluminum is important for light weighting of vehicles and use of aluminum can easily reduce the average car weight with more than 40 kg just through increased market penetration [9].

Aluminum metal matrix alloy requires some adjustments in its structure or physical properties in order to get an excellent range of functions. The addition of reinforcement to aluminum metal matrix alloy is the method to produce a composite that can improve properties such as tensile strength, wear resistance, thermal stability and electrical conductivity. Thus, the aluminum alloy industry, focuses are given to the manufacturing of aluminum metal composites to obtain enhanced of properties such as mechanical properties and thermal stability [10].

1.2. Metal Matrix Composites

The processing and developments of metal matrix composites (MMCs) have emerged as a class of materials capable of advanced structural, aerospace, automotive, electronic, thermal management and wear applications.

The performance advantage of metal matrix composites is their tailored mechanical, physical and thermal properties that include low density, high specific strength, high specific modulus, high thermal conductivity, high abrasion and wear resistance [11]. Another advantage of using metal matrix composite is that they can be plastically deformed and strengthened by a variety of thermal and mechanical treatments.

For the fabrication of metallic matrix composites several type of manufacturing methods is employed. The choice of appropriate manufacturing method highly alters the resulting composite microstructure, dispersion of reinforcement, interfacial bonding between the matrix phase and reinforcement particle and mechanical performances [12]. The manufacturing method to be employed is greatly based upon the type of reinforcement and metallic material. The major disadvantage behind producing aluminum matrix composites (AMCs) generally rests on the very expansive cost of production and reinforcement material. The economical and easy technique for production of composite materials is very important for elaborating their applications and properties [13]. Stir casting techniques are attracting researchers due to economical and easy method of processing. Several processing techniques which have shown to be comparatively expensive and complex in operation like in-situ casting, powder metallurgy and squeeze casting are also available. This study gives the production of aluminum centered metal matrix composite (AMCs) by using stir casting technique [14].

1.3. Statement of problem

This research is aimed to improve mechanical properties (tensile strength and hardness) between eggshell powders in aluminum (AA6063) alloy matrix composites made by stir casting method. The research carried out earlier by other researchers is mainly focused on the varying eggshell weight percentage during stir casting process to improve the mechanical properties. Thus, the focus of this thesis is to identify the significant factors (parameters) from stir casting process that affects the tensile strength and hardness of AA6063/eggshell powders composite. Another stir casting process parameters such as stirring speed, stirring time and stirring temperature are not so far considered when eggshell is used as reinforcement.

In order to solve this problem by applying Taguchi's method an experiment is conducted by identifying the significant factors from the stir casting process that affects the mechanical properties of composite. This problem can be resolved by development of optimum stir casting process parameters. The optimized stir casting process parameters used to reduced gas voids, defects such as porosities which lead to an improvement in the composite characteristics. As a result, a better understanding of stir casting process should be achieved in order to improve tensile strength and hardness of composite. The proposed work is aimed to fulfill this problem.

1.4. Objective of the study

1.4.1. General objective

The general objective of this thesis is to fabricate and experimental analyze the mechanical properties of Aluminum (AA6063) alloy/eggshell composite for different experimental conditions.

1.4.2. Specific objective

The specific objective of this study are:

- ❖ To prepare calcined (carbonized) eggshell powder
- ❖ To fabricate the composite materials for different eggshell composition
- ❖ To optimize stir casting process parameters
- ❖ To investigate microstructural changes for different eggshell composition
- ❖ To conduct Tensile strength and hardness testing of prepared composite

1.5. Scope of the study

Development of aluminum metal matrix composites starts with choosing an appropriate class of Al alloys as the matrix according to the desired applications. Aluminum alloys are preferred engineering material for many applications because of their strength, lower weight, excellent thermal conductivity properties. Among several series of aluminum alloys that are available non-heat treatable AA6063 alloy is selected for this study, because of its excellent properties such as strength to weight ratio, weldability, low density, ductility, good corrosion resistance, good formability and workability. AA6063 alloy is primarily composed of silicon and magnesium as the alloying agent. Silicon is primarily responsible for good castability (high fluidity, low shrinkage) and magnesium provides substantial strengthening and improvement of the work hardening characteristics of aluminum. Silicon combine with magnesium to form the hardening phase Mg_2Si that provides the strengthening.

Selection of the reinforcement materials should follow a criterion, including density, hardness, thermal compatibility with matrix material (wettability, reaction with matrix), availability, cost, renewability and biodegradability etc. In this study waste eggshell is selected as reinforcement material. The reason of selecting waste eggshell as a reinforcement material is derived from its $CaCO_3$ content, renewability and availability in bulk quantity with lightweight, minimizing waste eggshell, being economic and environmental friendly. The most effective chemical modification (carbonization) will be done on eggshell powder to make it more reactive with aluminum alloy.

Fabrication of eggshell powder in aluminum (AA6063) alloy can be processed by stir casting technique to produce applicable composite. Because stir casting is very efficient, simple and cost effective method of manufacturing aluminum metal matrix composites and also it is most suitable for mass production. Once the composite is fabricated, specimens are formed for microstructure study, tensile and hardness tests.

1.6. Significance of the study

The main benefits of this aluminum (AA60630 alloy/eggshell composite are;

- ❖ Utilization of finite source of non-renewable calcium carbonate
- ❖ Protect eggshell from pollute the environment
- ❖ To reduce the cost and the weight of composite and
- ❖ To produce composite materials for different applications.

1.7. Organization of the study

This research paper is composed of five chapters. The first chapter deals with the introduction, metal matrix composites, statement of problem, objective of the study, scope of the study, significance of the study and Organization of the study have been discussed. In the second chapter, literature review in the area of eggshell as reinforcement material, aluminum alloy as matrix material and their composite mechanical properties have been discussed. The third chapter deals with preparation and calcination of eggshell and its experimental procedure with aluminum (AA6063) alloy to make composite material. Testing, data collection, discussion, analysis and interpretation have been discussed in the fourth chapter. Finally, conclusion, recommendation and future works have been discussed in fifth chapter.

CHAPTER 2: LITERATURE REVIEW

2.1. Introduction

Composite materials appear everywhere in life, both man-made and biologically produced (like mammalian bones). The purpose of a composite material is to alter and improve the properties of the matrix material, by the addition of some second material with very different chemical and structural properties. An example of an important area of composite research is metal matrix composites, in which a metallic material is modified through the addition of a reinforcement. The reinforcement may be in the form of particles or fibers. The goal is to supplement the desirable properties of the metal, such as ductility, by the addition of a reinforcement which can improve the performance of the material in its final application. Common goals in the creation of new metal matrix composites are to achieve increased strength, hardness, and prevent corrosion.

The need of materials with optimum properties and low cost for various engineering applications has paced researchers in area of metal matrix composites. Aluminum, both in pure and alloy form are a suitable material for many engineering and industrial applications. Thus, the physical, chemical and mechanical properties of aluminum have fascinated researchers to explore its potential. In aluminum matrix composites, the extensively used reinforcement is in particulate form and is widely employed in automotive and aerospace industries [15].

Therefore, a tremendous demand for utilization of aluminum matrix composites in all sectors because of its better performance, economic and environmental benefits. Furthermore, aluminum alloy matrix composites are considered as the most popular among all metal matrix composites and are required for many engineering applications. Aluminum matrix composites (AMCs) have the following major advantages when compared to unreinforced materials [15];

- ❖ Reduced density (less weight)
- ❖ Higher strength
- ❖ Thermal/heat management
- ❖ Enhanced and tailored electrical performance
- ❖ Improved abrasion and wear resistance
- ❖ Improved stiffness
- ❖ Improved high temperature properties
- ❖ Controlled coefficient of thermal expansion

2.2. Aluminum Alloys and its Mechanical Properties

Aluminum is the most abundant metal in the Earth's crust, and the third most abundant element, next oxygen and silicon [16]. The mechanical properties provided by aluminum and its alloys make aluminum one of the most versatile, economical and attractive metallic materials for a broad range of uses. Aluminum and aluminum alloy are gaining huge industrial significance because of their combination of mechanical and physical properties over the base alloys [17].

Pure aluminum (99.95%) strength is too low to be used structurally. But commercially pure aluminum (99.2%) containing slight amounts of silicon, iron, magnesium and manganese is nearly twice as strong. Working and alloying has the effect of improving the strength of commercially pure aluminum without affecting its light weight property. Aluminum has a density of only 2.7 g/cm^3, approximately one-third as much as steel (7.83 g/cm^3). Aluminum resists the kind of oxidization that causes steel to rust away. Appropriately alloyed and treated, aluminum can resist corrosion by water, other environmental factors, chemical and physical agents [18].

2.3. Applications of aluminum alloys

Aluminum offers weight saving because of its light weight. The softness of this metal can restrict its use for the engineering purposes. Aluminum alloy displays excellent electrical and thermal conductivity, but specific alloys have been developed with high degrees of electrical resistivity. Aluminum is often selected for its electrical conductivity, which is nearly twice that of copper on an equivalent weight. The thermal conductivity of aluminum alloys, about 50 to 60% that of copper, is advantageous in heat exchangers, evaporators, electrically heated appliances, utensils, and automotive cylinder heads and radiators [19]. Aluminum is non ferromagnetic, a property of importance in the electrical and electronics industries. Aluminum is also non-toxic used in containers for food and beverages. It has an attractive appearance in its natural, which can be soft and bright or shiny [20].

Aluminum alloys are economical in many application areas. They are used in the automotive industry, aerospace industry, in construction of machines, appliances, and structures, cooking utensils, covers for housings for electronic equipment, pressure vessels etc. [21]. Aluminum is used in the automotive industry to produce a variety of parts. The distribution of aluminum in a typical car, and the percentage of those parts that are cast or wrought aluminum alloys, are given in Table 2.1.

Table 2.1: Distribution of cast and wrought aluminum in automobile in 2010 [22].

Area of car	Portion of total aluminum used in car (%)	Cast aluminum alloy (%)	Wrought aluminum alloy (%)
Engine Parts	50	90	10
Chassis	30	90	10
Body	15	20	80
Interior Fittings	5	40	60

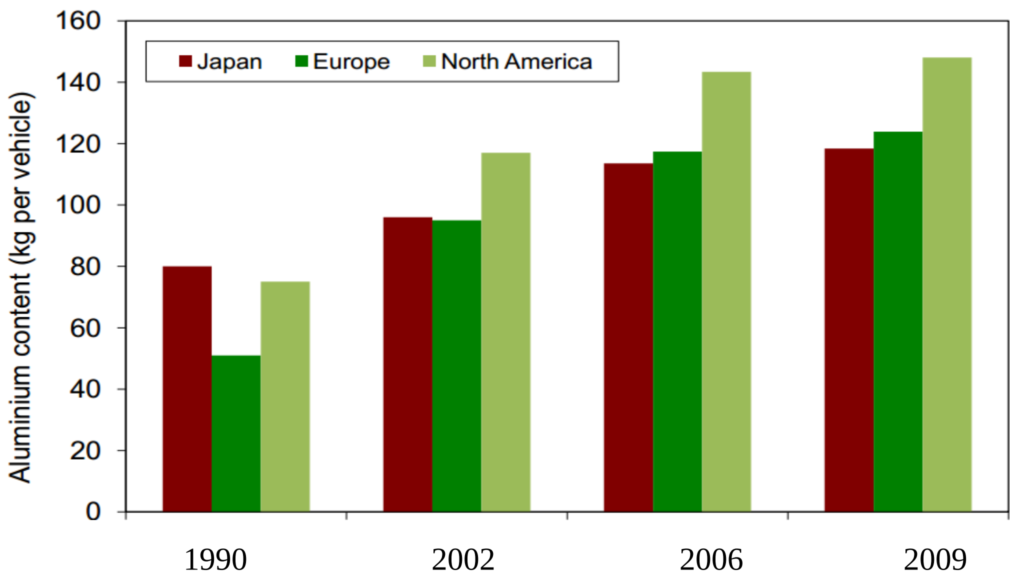

Figure 2.1 Average aluminum content in vehicles for North America, Europe and Japan [23].

2.4. AA6XXX Series Aluminum Alloys and its Designation

The strengthening of Al is principally done by alloying it with elements like Mg, Si, Cu, Zn, Mn, etc. and processing its alloys. Generally, a four-digit numerical designation is used to specify aluminum and its alloys, where first digit denotes the principal alloying element except AA1XXX series, which stands for pure aluminum. The second digit stands for the changes to impurity limits. The minimum % of Al (AA1XXX series) and different aluminum alloys in the group (for other series) are represented by the third and fourth digits [24]. Table 2.2 presents the designation of wrought aluminum and its alloys.

Table 2.1: AA designation of wrought aluminum and its alloys [25].

Alloying elements	Series designation
Pure aluminum	AA1XXX
Copper	AA2XXX
Manganese	AA3XXX
Silicon	AA4XXX
Magnesium	AA5XXX
Magnesium and silicon	AA6XXX
Zinc	AA7XXX
Lithium	AA8XXX

The castings Al and its alloys are designated by a four-digit number with the incorporation of a decimal point. Number 1 in the first digit stands for aluminum with high purity. The second and third digits are used for minimum Al %. The fourth digit is used after decimal point and it is 1 for cast ingot and 0 for casting. Table 2.3 presents the designation of cast aluminum and its alloys.

Table 2.2: AA designation of cast aluminum and its alloys [25].

Alloying elements	Series designation
Pure aluminum	AA1XX.X
Copper	AA2XX.X
Silicon, with copper or magnesium	AA3XX.X
Silicon	AA4XX.X
Magnesium	AA5XX.X
Zinc	AA7XX.X
Lithium	AA8XX.X

2.5. Mechanical Properties of Aluminum (AA6063) Alloy

Aluminum is frequently alloyed in order to increase strength. AA6063 alloy is an aluminum alloy with alloying elements magnesium and silicon. The aluminum association controlling its composition standard. It has various good mechanical properties, weldability, very good corrosion and heat treatable [26]. The typical applications of AA6063 alloy are architectural and building products, railings, door and window frames, pipe and tube for irrigation systems, ladders etc.

Density of AA6063 alloy is 2.7 g/cm^3 [27]. Heat treatment process affect the mechanical properties of AA6063 alloy. Un-heat-treated AA6063 alloy has maximum tensile strength no more than 130 MPa, and has no specified yield strength. The major elements of AA6063 alloy are Si and Mg [28]. The chemical composition of aluminum (AA6063) alloy is given in Table 2.4.

Table 2.3: Chemical composition of aluminum (AA6063) alloy [29]

Elements	Cu	Mg	Si	Fe	Mn	Ni	Zn	Pb	Ti	Al
Percentage	0.2	0.5	0.6	0.3	0.4	0.1	0.1	0.1	0.2	97.5

The physical properties of aluminum (AA6063) alloy is given in table 2.5.

Table 2.4: Typical physical properties of AA6063 alloy [29]

Melting Range	580-660^{0}C
Density	2.7 g/cm^3
Thermal Conductivity	197 W/m^{0}k
Thermal expansion coefficient	24×10^{-8}
Electrical conductivity	49.3 IACS
Electrical resistivity	0.035 microhm m
Modulus of elasticity	69 GPa

The mechanical properties of aluminum (AA6063) alloy is given in table 2.6.

Table 2.5: Mechanical Properties of AA6063 alloy [29]

Density (kg/m^3)	2700
Poisson Ratio	0.33
Vickers Hardness (VHN)	77
Tensile strength(MPa)	90

Silicon (Si): Silicon is the most important alloying element used in majority of aluminum casting alloys [30]. It is primarily responsible for good castability (high fluidity, low shrinkage) and low density (2.34g/cm^3) which can be reducing total weight of cast component. Si has very low solubility in aluminum alloy and precipitates as virtually pure Si which is hard and improve the abrasion resistance.

Magnesium (Mg: It can provide good corrosion resistance, weldability and extremely high Strength to aluminum [30]. Silicon combine with magnesium to form the hardening phase Mg_2Si

that provides the strengthening. The proper Mg addition amount is very important to enhance the mechanical properties of the graphite particles reinforced aluminum alloy matrix composites, uniformly distribution of hard particles and reduction of friction coefficient [31].

2.6. Reinforcements

Reinforcements are used in different amount with the composite based on the size required in order to improve mechanical and physical properties of metal matrix materials [32]. Reinforcement can be classified as inorganic and organic reinforcement. They can be further divided based on volume, size ratio, shapes, and their enhancements in composites [33]. For instance, calcium carbonate is falls in the category of inorganic reinforcement while eggshell falls in the category of organic reinforcement.

2.6.1. Organic Reinforcements

Organic reinforcement can be produced by recycling waste material from plants and animals. The organic reinforcement has low density, need low cost to produce and available abundantly. Therefore, it is suit to replace the inorganic reinforcement for composite materials in many applications [34].

2.6.1.1. Structure and Composition of Eggshell

Chicken egg consists of 60% albumen, 30% yolk and another 10% of eggshell and membrane [35]. The different parts of chicken egg are presented in the Figure 2.2. Eggshell consists of eggshell membrane and calcified eggshell filler. Eggshell is a combination of both inorganic and organic components. The main component of eggshell is calcium carbonate ($CaCO_3$) in the form of calcite which has a density of 2.710 gcm^{-3} [36]. The other components are 1 wt.% magnesium carbonate, 1 wt.% calcium phosphate, and organic materials such as type X collagen, sulfated polysaccharides and other proteins about 3 wt.% [37].

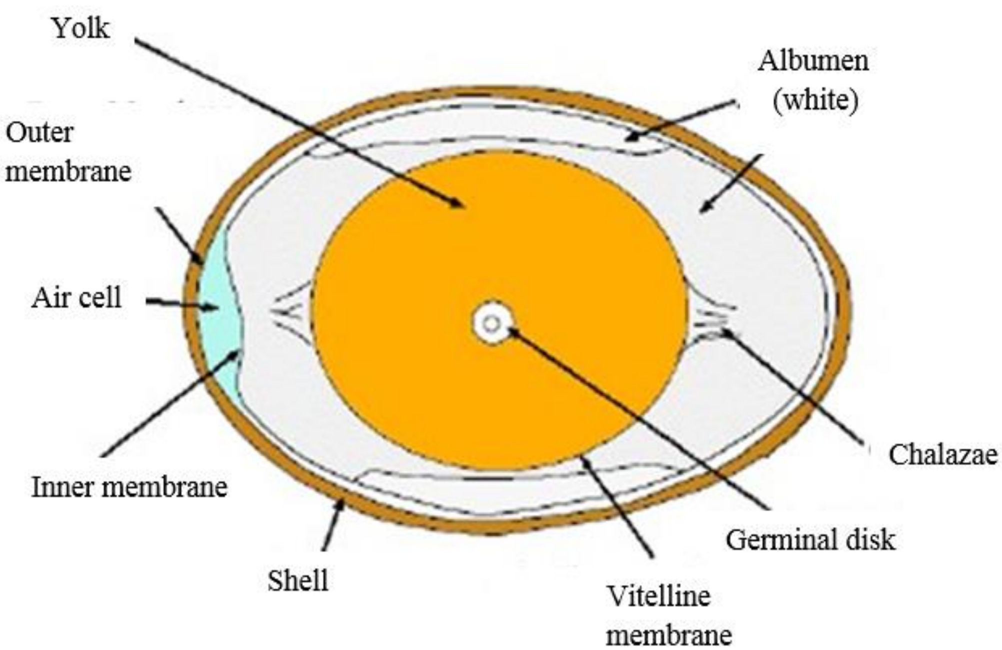

Figure 2.2 Schematic of different parts of egg structure [38]

The eggshell which forms the outer crust of an egg is a natural bioceramic. The structure of the eggshell and membrane is now very well understood due to scanning electron microscopy and micro focus X-ray scattering techniques [39]. The calcified portion (consisting of calcium carbonate crystals) of the shell which precedes the outer membrane can be divided into three layers; the mammillary layer, the palisade layer and the vertical crystal layer [40].

The mammillary layer (70 µm thick) which forms the inner most layer of the calcified portion of the eggshell penetrates the outer membrane by means of numerous carbonate cones. The initiation of the formation of calcium carbonate crystals takes place at the mammillary layers, which are organic cores deposited during the egg formation [40].

The palisade layer (200 µm thick) lies above the mammillary layer and forms the major portion of the calcified layer of the eggshell. The palisade layer gives way to the vertical crystal layer [40].

The vertical crystal layer which is about 8 µm thick is a very thin layer and consists of the upper most part of calcite crystals which provides a surface for the formation of the cuticle [41]. The cuticle is the outer most water insoluble layer of the eggshell (10 – 30 µm thick). The layer is largely an organic layer with protein contents as high as 90% and with a high content of cystine, glycine, glutamic acid, lysine and tyrosine [41].

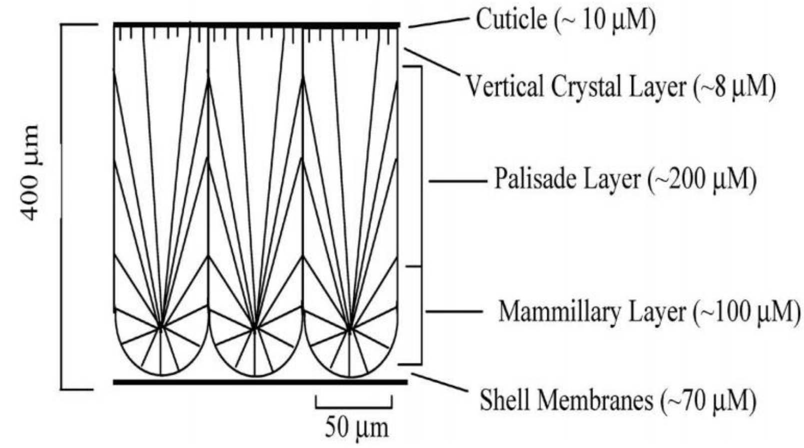

Figure 2.3 Schematic diagram of the structure and different layers within the eggshell [42]

2.6.2. Inorganic Reinforcements

The most widely used inorganic reinforcements in the composite industry are silicon carbide (SiC), calcium carbonate (CaCO3) particles and boron nitride, and layered silicates. They are added to enhance the properties of metal matrix composites [43]. One of the major classes of inorganic reinforcement are mineral fillers. The filler particle with lower density decreases the density of a product and then lowers the price [44]. Normally, fine mineral particles are produced from natural resources through grinding and sieving process to obtain a specified particle size. The natural resources for mineral filler production are however limited in quantity and take very long time to regenerate. Among those mineral filler Calcium carbonate is one of the most popular reinforcement used in the aluminum alloy industry [44]. If they are continually consumed with a filler production growth rate of 5% per annum , the raw materials for filler production will be finally short in the near future [45]. The issues of sustainable development and environmental friendly materials are currently great interest. Many attempts have been done to employ mineral fillers from bio-based and renewable sources including rice husk ash (RSH) [46], and cuttlebone [47].

2.7. Calcination (carbonization) of Eggshell

The waste eggshell is applied as particulate mineral filler only in the form of calcium carbonate. Actually, the waste eggshell is also a renewable raw material for calcium carbonate filler.The calcination of eggshell at the treatment temperature of 700 ^{o}C, found that the major component of the calcined eggshell (CES) was CaO [48]. It was also reported that calcination of eggshell above 500 ^{o}C would help removing organic membranes and matrix protein [48]. Preparation of calcium hydroxide from waste eggshell is possible by calcination of the eggshell above decarbonation temperature of calcium carbonate, approximately 800 ^{o}C, following with atmospheric exposure. As a matter of fact, calcium oxide itself as a main composition of CES. The calcined eggshell (CES) is able to turn into calcium carbonate upon atmospheric exposure [48].

Eggshell without eggshell membrane and its particle size of less than 200 μm was used to prepare carbonized eggshell at 200-900 °C for 1 hour and identify its crystal structure by X-ray diffraction (XRD) [50]. They found that the crystal structure of calcined eggshell was identified as calcite ($CaCO_3$) only at the treatment temperature from 200 °C up to 600 °C. The density of eggshell was reduced from 2.71gcm^{-3} to 2 gcm^{-3} after eggshell was calcined at the treatment temperature from 200 ^{o}C to 600 ^{o}C for 2 hours [51]. However, the calcium carbonate phase decreased and CaO appeared as the eggshell was treated above 700 °C. In addition, it was reported that the treatment of eggshell at 900 °C for 1 hour led to the complete phase transformation from $CaCO_3$ to CaO. The calcination of eggshell below 600 °C for 2 hour did not cause to the formation of CaO. However, the eggshell was calcined at 700 °C for 2 hour obtained $CaCO_3$ as a major component and CaO as a minor component [52].

2.8. Mechanical Properties of Aluminum Metal Matrix Composites

Composites are man-made or naturally occurring materials made from two or more constituent materials with different physical and chemical properties that remain separate and distinct within the finished structure. The bulk material forms the continuous phase that is the matrix (e.g. metals) and the other acts as the discontinuous phase that is the reinforcement (e.g. fibers, whiskers, particulates) [53]. Composite materials are gaining wide spread acceptance due to their high strength to weight ratio [54]. Mechanical properties were affected by selection of the weight percentage, size, and distribution of the reinforcing particles in the matrix [55].

AMMC are the competent material in the industrial world due to its excellent mechanical properties [56]. The aluminum matrix is getting strengthened when it is reinforced with the hard ceramic particles like B_4C, SiC, MoS_2 and Al_2O_3 etc. Aluminum alloys are still the subjects of studies, as their low density gives additional advantages in several applications.

Aluminum metal matrix composites shows better improvement in hardness with the increasing in percentage of reinforcement and decrease in density [57]. Mechanical properties (hardness, tensile strength and yield strength) of composites were increased with increasing wt.% addition of reinforcements [58]. Aluminum alloy AA6063 is available in a wide range of structural shapes, sheet and plate products. The development of a new material with greater mechanical properties, without much compromising on the strength to weight ratio led to the development of the metal matrix composites [59].

Mechanical properties of metal matrix composites depend on the wettability property of metal matrix composites. If wettability between matrix and reinforcement particles is good, then mechanical properties is improved [60]. The wettability results showed that Al and SiC developed good wettability at interfaces of metal matrix composites. Basically, silicon carbide (SiC), alumina (Al_2O_3), boron carbide (B_4C) and $CaCO_3$ are the four traditional ceramic particles which are used as a reinforcement material in aluminum alloys. These reinforcement particles are available in the market. The increasing demand for low cost reinforcement stimulated the interest towards production and utilization of using waste products from industry as reinforcement since they are readily available or are naturally renewable at affordable cost. The mechanical properties (hardness and tensile strength) of aluminum alloy are discussed below.

2.8.1. Tensile strength

The tensile test is the most widely used test to determine the mechanical properties of composite materials. In this test, a sample of material is pulled until it fractures. During the test, the specimen elongation and applied force is measured. Strain and stress are calculated from these values, and are used to construct a stress-strain graph. From this graph, the elastic modulus and yield strength are determined. The yield strength (YS) is defined as the maximum stress at which a material begins to deform plastically and is located above the elastic limit. The yield strength of a composite is best defined by a set deviation from linear-elastic behavior. The highest load in the tensile test gives the ultimate strength. The ultimate tensile strength (UTS), or tensile strength is defined as the maximum stress experienced by the sample before failure (before the neck formation). After fracture, the final length and cross-sectional area of the sample are used to calculate the elongation and percent reduction of area respectively. The following formulas were used to determine ultimate tensile strength (UTS) [61].

$$\text{Ultimate Strength} = \frac{Maximum\ load}{Original\ cross\ sectional\ area} \tag{2.1}$$

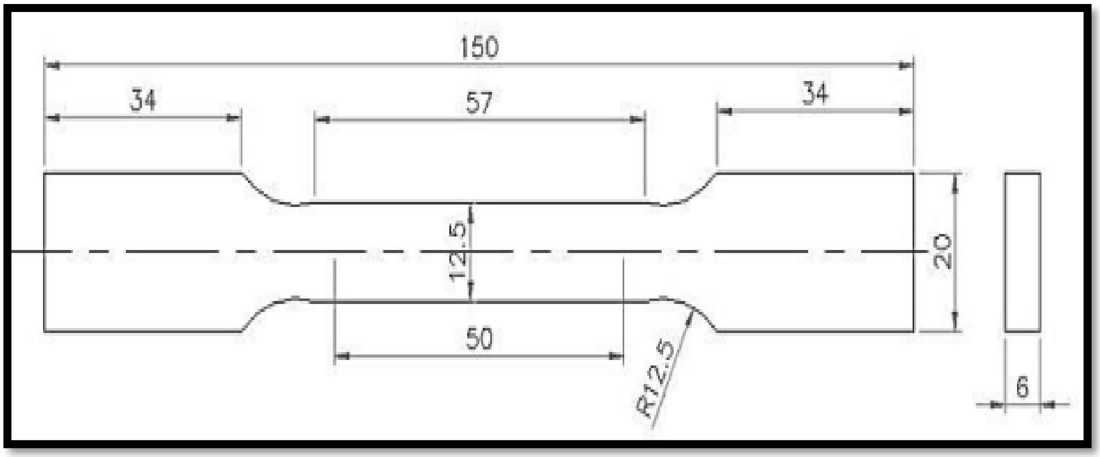

Figure 2.4 Tensile test specimen as per ASTM E8 standard [61]

2.8.2. Hardness

Hardness is the property of a material to resist permanent indentation. The hardness of composite depends on the hardness of the reinforcements and matrix materials [62]. The type of material and expected hardness will determine test method. There are several methods of measuring hardness. The Rockwell Hardness number (HRB), the Brinell Hardness Number (BHN) and the Vickers Hardness number (VH) are used to measure the hardness of the materials prepared in specimen form.

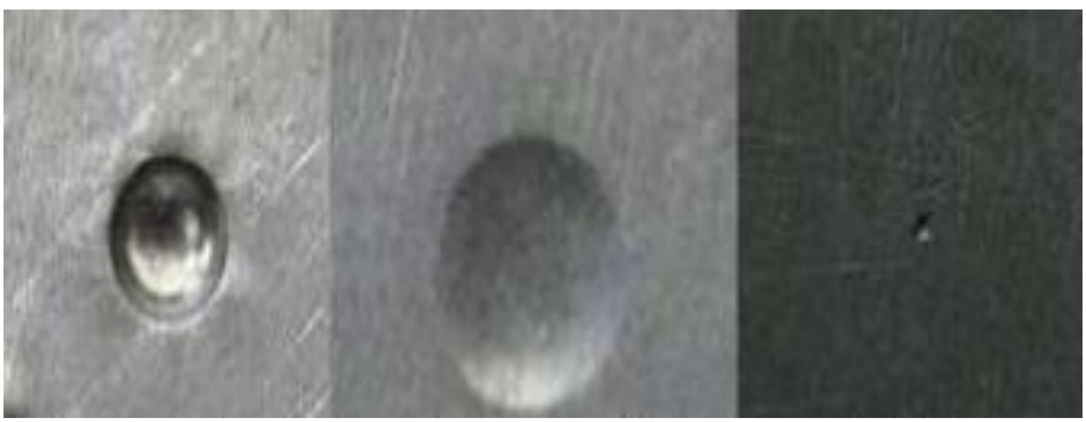

Figure 2.5 The indentation of Rockwell, Brinell, Vickers hardness test respectively [63]

Rockwell Hardness Test: The Rockwell hardness test determines the hardness by measuring the depth of penetration of an indenter under a large load compared to the penetration made by a preload [64]. The determination of the Rockwell hardness of a material involves the application of a minor load followed by a major load. Release the major load and the dial indicator gives the Rockwell hardness number (HRB). Materials such as hardened bearing steels have small grain size and can be measured using the Rockwell scale due to the use of diamond indenters and high PSI loading.

Brinell Hardness Test: The indentation hardness of materials through the scale of penetration of an indenter, loaded on a material test [65]. Materials such as cast irons and powder metals will need a much larger indenter such as used with Brinell scales. The value of the Brinell Hardness Number (BHN) is obtained by applying the following formula.

$$\text{BHN}=\frac{2p}{\left(\pi D-\sqrt{(D^2-d^2)}\right)} \tag{2.2}$$

Where P = Load applied, D = Diameter of indenter, d = Diameter of indentation

Vickers Hardness Test: The Vickers test method principle is similar to the Brinell hardness testing method. The principle in this test is that a defined shaped indenter is pressed into the material. The indenting force is applied for a certain amount of time [66]. The resulting indentation diagonals are measured and recorded. The hardness number is calculated by dividing the force by the surface area of the indentation. As mentioned previously, the principle of the Vickers test is similar to the Brinell test, but the Vickers test is operated with different forces and indenters [67]. Very small parts or small sections may need to be measured on a microhardness tester using the Vickers or Knop Scale. The square pyramidal diamond indenter is forced under a predetermined load ranging from 1 to 129 kgf into the material to be tested. After the forces have attained static equilibrium conditions, the force remains applied for a specific time (10 to 15 seconds for normal test times) and is then removed.

The resulting unrecovered indentation diagonals are measured and averaged to give the value in millimeter. These length measurements are used to calculate the Vickers hardness number (VH). The Vickers Hardness Number (VH) is obtained by applying the following formula [67].

$$\text{HV}=\frac{2P\sin(\frac{136}{2})}{d^2}=\frac{1.8544P}{D^2} \tag{2.3}$$

Where, P is the indentation load in kgf and d is the average diagonals of indentation in mm. The Vickers indenter has included face angles 136˚.The load applied was 5 kgf for a duration of 15 seconds.

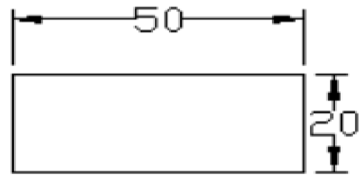

Figure 2.6 Specimen for hardness test

2.8.3. Microstructural of Metal Matrix Composites

Microscopic analyses of the matrix metal and composite specimens were performed by optical microscopy. An image analyzer was used to examine the distribution of the reinforcement particles within the aluminum alloy. The mechanical properties of any particle reinforced metal matrix composites depend on the particle distribution and particle matrix bonding. It is therefore, necessary to conduct a microscopic analysis on the new material in order to gain better understanding of its microstructural characteristics. The polished specimens were cleaned with acetone and etched with (Methanol (25ml) + Hydrochloric (25ml) + Nitric Acid (25ml) + Hydrofluoric acid 1 drop) [68].

2.9. Fabrication Methods of Metal Matrix Composites

For the fabrication of metallic matrix composites several type of manufacturing methods is employed. The selection of appropriate manufacturing method highly alters the resulting composite microstructure, dispersion of ceramic agents, interfacial bonding between the matrix phase and reinforcement particle and mechanical properties [69]. The manufacturing method to be employed is greatly depend on type of reinforcement and metallic material. The economical and easy technique for production of composite materials is very important for elaborating their applications and properties. Stir casting and modified stir casting (two step stir casting) techniques are attracting researcher’s due to economical and easy method of processing. Several processing techniques which have shown to be comparatively expensive and complex in operation like in-situ casting, powder metallurgy and squeeze casting are also available.

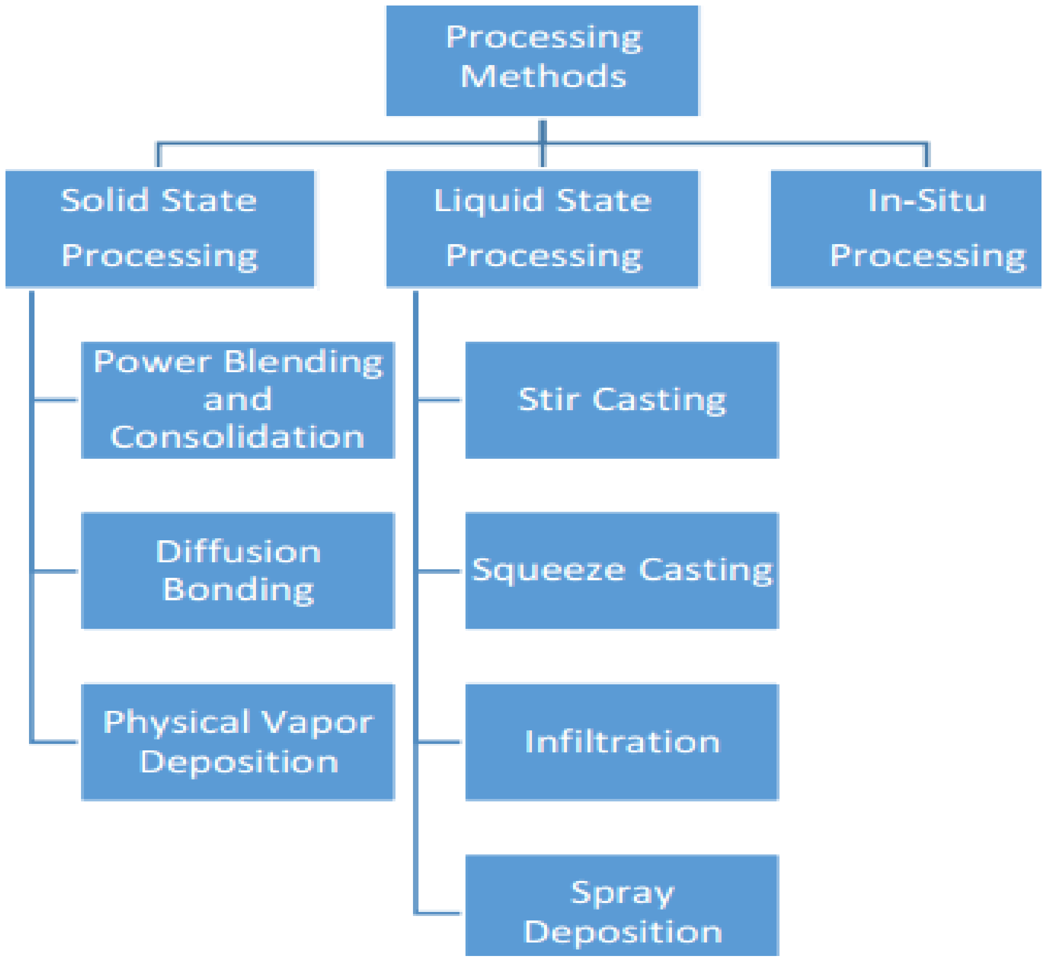

Figure 2.7 processing of metal matrix composites [70]

In the casting process, high temperature melt is used. High temperature often promotes the chemical reaction between the melt and the reinforcement materials. The reaction leads to the degradation or disappearance of the reinforcements. The expected properties of the composite would not be obtained if this reaction occurs. So in order to obtain metal matrix composite with good characteristics special techniques are required. Among various manufacturing methods stir casting is generally accepted because of low cost, little damage to reinforcement and stir cast components are not restricted by its size and shape [70]. It also possesses advantages like simplicity, flexibility and applicability to large quantity production [71].

Fabrication methods used for production of MMCs should ensure uniform distribution of reinforcement in matrix phase. The non-uniform distribution is due to density differences between the reinforcement particles and the metal melt. Manufacturing methods should also ensure proper wettability of reinforcement in matrix materials. Wettability can be defined as the ability of a liquid to spread on a solid surface. It also describes the extent contact (bond) between a liquid and a solid.

Table 2.6: Comparative analysis of different techniques of fabrication of MMC [72].

Method	Range of shape and size	Metal yield	Damage to reinforcement	Cost
Powder metallurgy	Wide range, restricted by size	High	Reinforcement fracture	Expensive
Squeeze casting	Limited by perform shape up to 2 cm height	Low	Severe damage	Moderately expensive
Spray casting	Limited shape, large size	Medium	-	Expensive
Stir casting	Not limited by size	Medium	Little damage	Moderately expensive
Electromagnetic stirring	Not limited by size	High	No damage	Moderately expensive

2.10. Stir Casting

Stir casting is a liquid state method of composite materials fabrication, in which a dispersed phase is mixed with a molten matrix metal by means of mechanical stirring. In preparing metal matrix composites by stir casting method some of the factors that need considerable attention are [73];

- ❖ To achieve uniform distribution of the reinforcement material
- ❖ To achieve wettability between the two main substances
- ❖ To minimize porosity in the cast metal matrix composite
- ❖ Chemical reactions between the reinforcement material and the matrix alloy [74].

Introducing reinforcement particles to the stirred molten matrix sometimes will entrap impurities such as metal oxide and slag, which is formed on the surface of the melt. During pouring, air envelopes may form between particles, which can alter the interface properties between particles and the molten metal, and also retarding the wettability between them. A major concern associated with the stir casting process is the segregation of reinforcing particles which is caused by the floating or settling of reinforcement particles during melting and casting process. To prevent settling of particles a motor driven stirrer is used [75]. The final distribution of the particles in the solid depends on material properties and process parameters such as the wetting condition of the particles with the melt, strength of mixing, relative density, and rate of solidification. The distribution of the particles in the molten matrix also depends on the shape of the mechanical stirrer, stirring time, placement of the mechanical stirrer in the melt, melting temperature, and the characteristics of the particles added [76]. To create a good distribution of the reinforcement material in molten matrix a vortex method is used. In this method, after the matrix material is melted, it is stirred vigorously to form a vortex at the surface of the melt, and the reinforcement material is then introduced at the side of the vortex. The stirring is continued for a few minutes before the slurry is cast [77]. Figure 2.8 presents the stir casting procedure followed to produce composite materials.

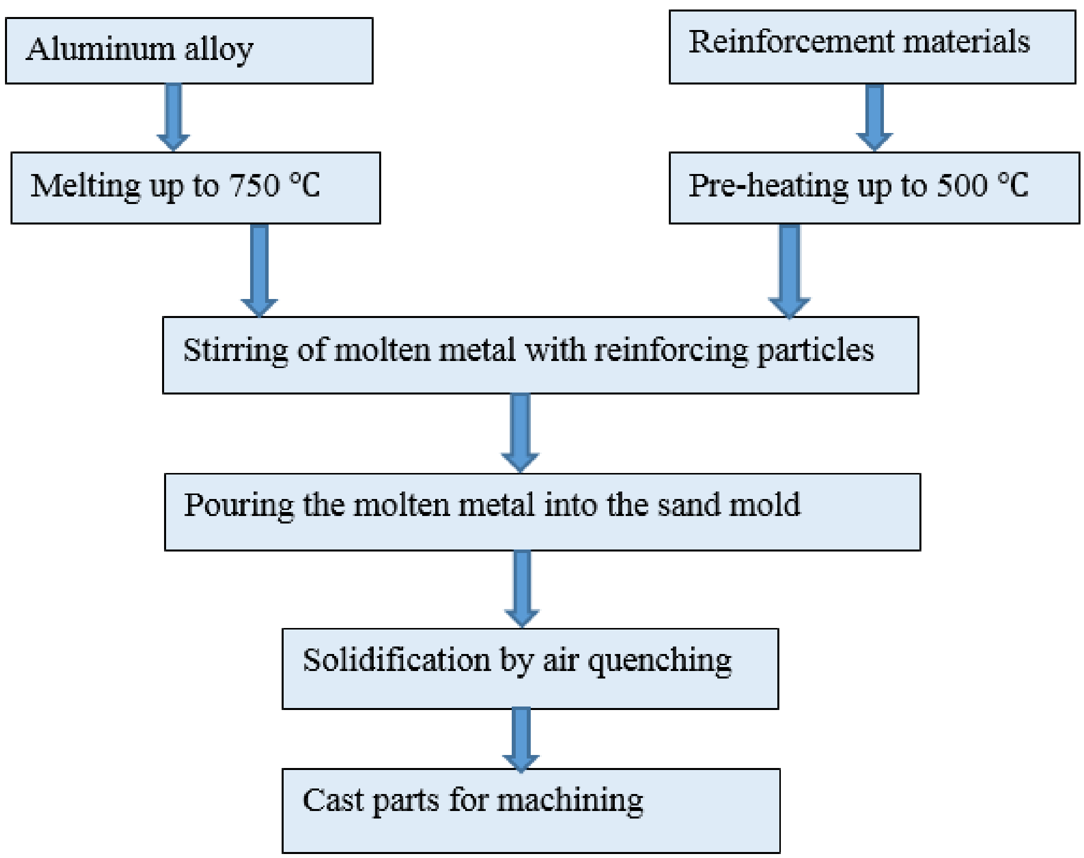

Figure 2. 8 Stir casting procedure flow diagram [78]

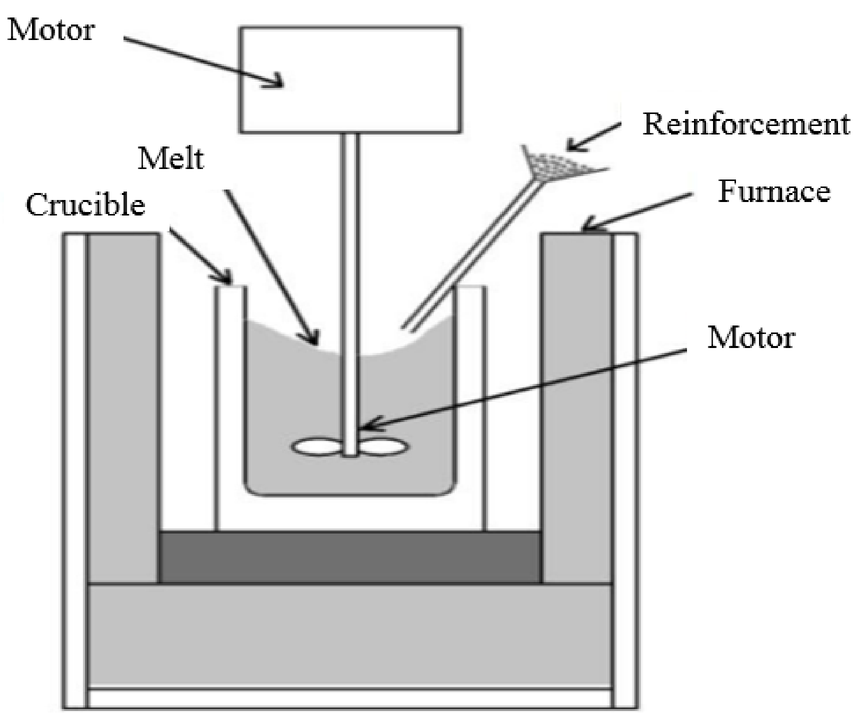

Figure 2.9 Stir casting setup [79]

The homogeneous dispersion of the reinforcing phase is a key challenge in the processing of composite materials in order to achieve a defect free microstructure. The reinforcement phase depending on size in the composite may be either in the particle form. Particulate reinforced composites comparatively have cheap material cost and suitability of manufacturing method over other reinforced composite and that is why they are more preferable over fiber reinforced composites.

Stir casting method is appropriate to produce composites up to 30 wt.% of particles reinforcement [80,81]. Many aluminum matrix composites with various matrix compositions, such as AA6063 [82], AA356 [83] and AA6061 [84] have been produced successfully using stir casting technique. A homogeneous distribution and strong bonding of reinforcement particles in the composite matrix is critical for executing a high strength. The uneven distribution and weak bonding can lead to failures in reinforcement rich and reinforcement free zones. In reinforcement free areas under an applied load, slip of dislocations and initiations of micro cracks can occur easily, eventually resulting in failure of the composite material. Agglomeration or clustering of reinforcing particles causing a weak bond formation, which can bring to reduce the mechanical properties.

2.10.1. Stir Casting Process Parameters and their Effects

For manufacturing of composite material by stir casting method, knowledge of its operating parameter are very essential. There are various process parameters if they properly controlled can lead to the improved mechanical properties in composite material [85,86]. The important process parameters are;

- Stirring speed
- Stirring temperature
- Reinforcement preheat temperature
- Stirring time (Holding time)
- Powder feed rate
- Blade design of stirrer

Stirring speed: Stirring speed is the most important process parameters as wettability is promoted by stirring i.e. bonding between matrix and reinforcement. The flow pattern of the molten metal is controlled by the stirring speed. It is logical to use the highest speed to avoid tearing. Research work suggested that the speed range between 200 and 600 rpm are used for many molten materials.

Stirring temperature: The viscosity of molten matrix is influenced by the processing temperature. The particle distribution in the matrix is subjective to the change of viscosity. When processing temperature is increased along with increasing stirring time, there is a decrease in the viscosity of liquid. Good wettability is obtained by keeping temperature at 750°C.

Reinforcement pre-heat temperature: In order to remove moisture or any other gases present within reinforcement, the reinforcement was preheated at 500 ^{o}C temperature for 30 minutes. The wettability of reinforcement with matrix is promoted by preheating [87].

Stirring time: Uniform distribution of the particles in the molten metal and perfect interface bond between reinforcement and matrix is promoted by stirring time [88]. In the processing of composite, the stirring time between matrix and reinforcement is considered as important parameter. Stirring time is 5-10 minutes in many case.

Reinforcement Feed Rate: To have a good wettability of casting the feed rate of powder particles must be uniform. Non-uniform feed rate promotes clustering of particles at some places which causes the porosity and inclusion defect [89].

Stirrer Design: The blade angle and number of blades are important factor which decides the flow pattern of the liquid metal at the time of stirring. The blade with angle 45°C and 60°C will give the near-uniform distribution. The stirrer is immersed until two third depth of molten metal. Stirrer blades play an important role in creating vortex of the molten metal. This vortex formation ensures boding characteristic of matrix and reinforcement phase.

2.11. Reinforcement and its effect on mechanical properties

Many reinforcements were used for fabricating the aluminum metal matrix composites but significant increment in the mechanical properties of aluminum metal matrix composites was achieved when a certain percentage was added in the aluminum matrix material. The ultimate tensile strength and hardness were improved by adding different low cost reinforcements. The different low cost reinforcement in aluminum metal matrix composite is given in Table 2.8.

Table 2.7: Reinforcement and its effect on mechanical properties

Matrix material	Reinforcement material	Weight percentage	Improved reported properties
Aluminum	Maize stalk Ash [90]	20	UTM 824.62Mpa Hardness-57.80HRC
Aluminum- 2014-T6	Fly ash (Al_2O_3) [91]	20	UTM-515 MPa Hardness-114 VHN
Al-Cu-Mg	Egg Shell [92]	12	UTM-112 N/mm^2 Hardness-80HRF
Aluminum	Rice Husk Ash[93]	9-12	UTM-180Mpa Hardness-80BHN
Aluminum	Bread fruit hull Ash [94]		UTM-220 N/mm^2 Hardness-88HRB
Al-356	Fly Ash [95]	30	High electrical conductivity
Al-356	Fly Ash [96]	12	High wear rate i.e. 8*10^{-3} mm^3/m
Aluminum	Coconut Shell Ash [97]	15	Wear rate at 10N -5*10^{-3}g/m and at 20N -2*10^{3}g/m

2.12. Casting Process Parameters and Their Effects

Casting is a metal object produced by allowing molten metal to solidify in a mold cavity. The shape of the object is determined by the shape of the mold cavity. Casting process involves many parameters such as melting temperature of matrix materials, the mold condition (temperature, moisture content, sand and type of binders used), pouring temperature, and the gating design (pouring speed and runner size), the casting size, the type of alloy. The variation in these process parameters can affect the solidification of the molten metal in the mold and this in turn can affect crystals' formation. It is established that to ensure uniform cooling of the molten metal in the mold cavity, the law of uniform flow must be maintained (i.e. constant volume per second flow of the molten metal during the filling of the mold cavity) [98]. The effect of mold temperature and pouring temperature on the mechanical properties of aluminum alloy produced through sand casting was discussed below.

Pouring temperature: A major role is played by the pouring temperature on the mode of solidification process. Low pouring temperature is associated with maximum grain refinement while higher temperature promotes columnar growth in many alloys. To ensure moderate metal flow and freedom from collapse and coarse structures, the pouring temperature must be sufficiently high. However, pouring temperature was varied within the range of 700-800 ^{0}C [99].

Mold Temperature: Porosity is the major defect in casting. In order to avoid porosity preheating of mold is good method. It helps in removing the entrapped gases from the molten metal to go into the mold. Mold is heated to 200^{o}C for one hour [99].

Modern production methods for casting include the use of sand-mold, metal-mold, die, and centrifugal castings. Castings produced using sand mold is known to have less microstructures depending on average size, distribution, shape of the molding, sand grains and the chemical composition of the alloy. These affect the surface finish, permeability and refractoriness of all the castings. The effect of using CO_2 process, metal mold, cement-bonded and naturally-bonded sand mold on the hardness, tensile and impact strengths of cast AA6063 aluminum alloy are investigated. The results show that there is significant increase in hardness of the alloy when naturally-bonded sand mold is used for its production over that of metal, CO_2 and cement molds.

2.13. Taguchi Design of Experiments

In the design of experiment (DOE), Taguchi method is commonly used because it is a problem–solving tool which can improve the performance of the product, process design and system. This method combines the experimental and analytical concepts to determine the most influential parameter on the result response for the significant improvement in the overall performance. Design of experiment is a technique of defining and investigating all possible conditions involving multiple factors, parameters and variables in an experiment. Taguchi design of experiment consists of orthogonal arrays to study all the parameters of the project with a minimum number of experiments [100].

2.14. Taguchi's Method Process Parameter Optimization

Taguchi technique is an optimization power tool for the design of quality system. It introduced an integrated that is simple and efficient to find the optimum range of design for quality performance and computational cost [101]. Process parameter optimization is the key step in the Taguchi method high quality without increasing cost. This is because optimization of process parameters can improve quality and the optimal process parameters obtained from the Taguchi method are insensitive to the variation of environmental conditions and other noise factors.

A large number of experiments have to be carried out when the number of the process parameters increases. To solve this task, the Taguchi method uses a special design of orthogonal arrays to study the entire process parameter space with only a small number of experiments. Using an orthogonal array to design the experiment could help the designers to study the influence of multiple controllable factors on the average of quality characteristics and the variations in a fast and economic way, while using a signal-to- noise ratio to analyze the experimental data could help the designers of the product or the manufacturer to easily find out the optimal parametric combinations. Taguchi method uses the signal-to-noise (S/N) ratio instead of average value of the experimental data. The S/N ratio reflects both the average and the variation of the quality characteristics [101].

2.15. Design Steps in Taguchi Method

Taguchi design of experiment consists the following steps [102]:

- ❖ Identify the main function, side effects, and failure mode
- ❖ Identify the noise factors, testing conditions, and quality characteristics
- ❖ Identify the objective function to be optimized
- ❖ Identify the control factors and their levels
- ❖ Select the orthogonal array matrix experiment

- Conduct the matrix experiment
- Analyze the data; predict the optimum levels and performance
- Perform the verification experiment and plan the future action

2.15.1. Taguchi method objective functions

Once the decision variables are established, objectives of the experiments should be defined. These objectives are defined as follows [102]:

Nominal-the-best: where the designer wants to achieve a particular target value, for example, a required output voltage of a circuit;

$$S/N=-10\log_{10}\frac{1}{n}\sum_{i=1}^{n}y_i^2 \tag{2.4}$$

Where y_i= observed response value and n = number of replications.

Smaller-the-better: where the designer wants to minimize the system response since quality decrease as the system response increases, some examples are the response time of a computer, or a radiation leakage from a microwave oven.

$$S/N = -10\log_{10}\frac{\mu^2}{\sigma^2} \tag{2.5}$$

Where μ = mean and $\mu = \frac{\sum_1^n y_i}{n}$, $y_i =$ observed response value

and σ = variance (standard deviation) and $\sigma^2 = \frac{\sum_1^n (y_i-\mu)^2}{n-1}$

Larger-the-better: where the designer wants to maximize the system response since quality increases with the system response. For example, tensile strength and hardness of aluminum alloy.

$$S/N = -10\log_{10}\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2} \tag{2.6}$$

Where y_i= observed response value and n= number of replications

Dr. Taguchi's Signal-to-Noise ratios (S/N), which are log functions is based on orthogonal array experiments which gives much reduced variance for the experiment with optimum settings of control parameters. Taguchi has used Signal-Noise (S/N) ratio as the quality characteristic of choice. S/N ratio is used as measurable value instead of standard deviation due to the fact that, as the mean decreases, the standard deviation also deceases and vice versa [103].

2.15.2. Parameter diagram (P- Diagram)

The P-diagram explains the product as an input/output system with different categories of factors influencing a certain quality characteristic referred to as output or response (y), respectively [104]:

Signal factors: are the parameters set by the user or operator of the product to express the target value for the response of the product. For example, the steering wheel angle is the signal factor that specifies the turning radius of an automobile.

Noise factors: are the potential sources of variation and cannot be controlled by the designer. Examples of noise factors are operator skill, outside temperature and outside humidity.

Control factors: are those parameters that can be specified freely by the designer. Each control factor can take multiple values, called levels. Examples are hardness, dimensions and weights.

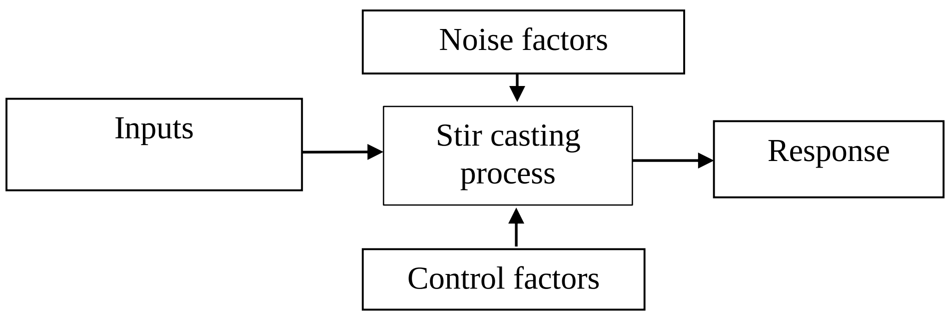

Figure 2.10 P-diagrams of stir casting processes [104]

The P-diagram helps engineers to view the product as a system and to recognize and distinguish the different factors affecting it.

2.15.3. Taguchi's Rule for Manufacturing

Taguchi realized that the best opportunity to eliminate variation is during the design of a product and its manufacturing process. Consequently, he developed a strategy for quality engineering that can be used in both contexts. The process has three stages:

- ❖ System design
- ❖ Parameter design
- ❖ Tolerance design

System design: This is design at the conceptual level, involving creativity and innovation.

Parameter design: The parameters (variables) are experimentally analyzed to determine how the product or process reacts to uncontrollable “noise” in the system; parameter design is the main thrust of Taguchi’s approach. Parameter design is related to finding the appropriate design factor levels to make the system less sensitive to variations in uncontrollable noise factors. In this way the product performs better, reducing the loss to the customer. Parameter design is sometimes called robustification. Robust parameter designs consider controllable and uncontrollable noise variables; they seek to exploit relationships and optimize settings that minimize the effects of the noise variables. Parameter design refers to the selection of control factors and the determination of optimal levels for each of the factors. The Taguchi method primarily focuses on parameter design [105].

Tolerance design: With a successfully completed parameter design, and an understanding of the effect that the various parameters have on performance, resources can be focused on reducing and controlling variation in the critical few dimensions.

2.15.4. Determining Orthogonal Array for Parameter Design

The effect of many different parameters on the performance characteristic in a condensed set of experiments can be examined by using the orthogonal array experimental design proposed by Taguchi. Once the parameters affecting a process that can be controlled have been determined, the levels at which these parameters should be varied must be determined.

Determining what levels of a variable to test requires an in-depth understanding of the process, including the minimum, maximum, and current value of the parameter. If the difference between the minimum and maximum value of a parameter is large, the values being tested can be further apart or more values can be tested. If the range of a parameter is small, then fewer values can be tested or the values tested can be closer together. The Taguchi method orthogonal array experimental design is a powerful tool for designing high quality systems. Knowing the number of parameters and the number of levels, the proper orthogonal array can be selected [105].

The selection of an appropriate orthogonal array (OA) depends on the total degrees of freedom of process parameters. Degrees of freedom are defined as the number of comparisons between process parameters that need to be made to determine which level is better and specifically how much better it is. Orthogonal Arrays (OA) provide a set of well balanced (minimum) experiments and Taguchi's Signal-to-Noise ratios (S/N), which are plot functions of desired output, serve as objective functions for optimization, help in data analysis and prediction of optimum results. Therefore, considering the above aspects, the experiments were designed using Taguchi method-based design of experiments methodology [106].

2.15.5. Run Experiment

There are different Taguchi analysis approaches that use quantitatively rigorous techniques such as analysis of variance (ANOVA), signal-to-noise ratios (S/N), and response charts.

2.15.6. Confirmation Experiment

Once the optimal levels for each of the factors have been determined, a confirming experiment with factors set at the optimal levels should be conducted to validate the earlier results.

2.20. Summary of Taguchi's Method

Taguchi started to develop new methods to optimize the process of engineering experimentation. He believed that the best way to improve quality was to design and build it into the product. He developed the techniques which are now known as Taguchi Methods. His concepts produced a unique and powerful quality improvement technique that differs from traditional practices. He developed manufacturing systems that were "robust" or insensitive to daily and seasonal variations of environment, machine wear and other external factors. The Taguchi approach to quality engineering places a great deal of emphasis on minimizing variation as the main means of improving quality. The idea is to design products and processes whose performance is not affected by outside conditions and to build this in during the development and design stage through the use of experimental design. The method includes a set of tables that enable main variables and interactions to be investigated in a minimum number of trials.

Process parameter optimization is the key step in the Taguchi method to achieving high quality without increasing cost. This is because optimization of process parameters can improve quality and the optimal process parameters obtained from the Taguchi method are insensitive to the variation of environmental conditions and other noise factors.

Therefore, this research proposes an effective stir casting process parameters (eggshell weight percentage, stirrer speed, stirring time and stirring temperature) for optimization approach to help the industry to obtain good quality product. Determining the process parameter settings for stir casting process greatly affects the quality of the stir casting product. Unsuitable process parameter settings can cause many production problems (e.g. many product defects, long lead times, large amounts of scrap, high production costs, etc.), increase the price competitive advantage, and reduced a company's profitability.

CHAPTER 3: MATERIALS AND METHODS

3.1. Materials and Equipment

3.1.1. Materials

AA6063 alloy was used as matrix metal and eggshell powder was used as reinforcement material. For composite development aluminum metal matrix (AA6063) alloy was purchased from B and C aluminum PLC company in Addis Ababa. The chemical composition of AA6063 alloy and its mechanical properties were shown in Table 3.1 and Table 3.2 respectively.

Table 3.1: Chemical composition of aluminum (AA6063) alloy in weight % [30]

Elements	Cu	Mg	Si	Fe	Mn	Ni	Zn	Pb	Ti	Al
Percentage	0.2	0.5	0.6	0.3	0.4	0.1	0.1	0.1	0.2	97.5

Table 3.2: Mechanical Properties of aluminum (AA6063) alloy [30]

Density	2.7 g/cm^3
Vickers hardness (VH)	77
Tensile strength	90 MPa

Figure 3.1 shows the photograph of AA6063 alloy chips looks spherical in shape.

Figure 3.1. Aluminum (AA6063) alloy powders

The chemical composition of eggshell powders is presented in Table 3.3.

Table 3.3: Chemical composition of eggshell in weight % [38,39]

Elements	$CaCO_3$	$MgCO_3$	$Ca_3(PO_4)_2$	Other organic
Percentage	95	1	1	3

3.1.2. Equipment

- Drilling machine
- Thermocouple
- Muffle furnace
- distilled water
- pestle and mortar
- sieve
- Stir casting process equipment
- Vickers hardness tester machine
- Universal tensile test machine
- Optical microscopy

3.2. Preparation of Eggshell Powder

The chicken eggshells (ES) were collected randomly from local environment such as households, poultry farming organizations, restaurants and hotels. The collected eggshells were washed for several times with distilled water. Membrane and shell were carefully separated by manual peeling. Then the chicken ES was made to dry under sun for 48 hours and smash into small pieces using a pestle and mortar. The dried ES has grinded into ES powder and the obtain powder was passed through sieve of require size (10-20µm) to obtain particles with uniform size distribution [92]. The photographs of uncarbonized eggshell and the membrane removed from the inner surface of eggshells were shown in figure 3.2.

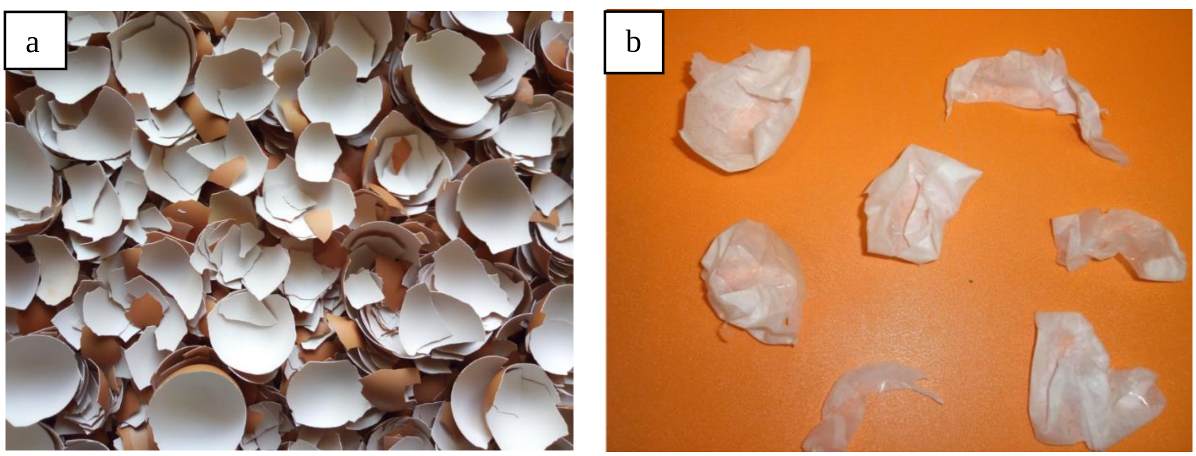

Figure 3.2 (a) uncarbonized eggshell, (b) Peeled eggshell membranes

Figure 3.3 shows the procedure followed to produce calcined (carbonized) eggshell powders.

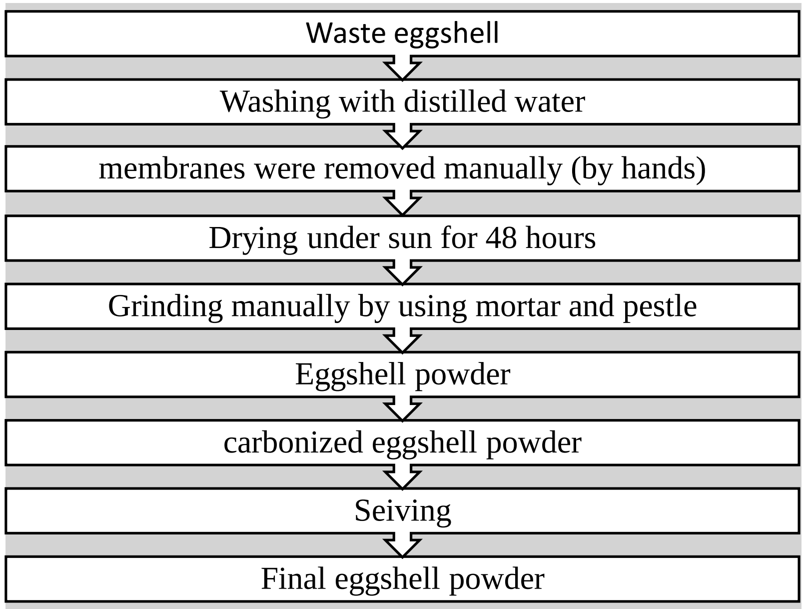

Figure 3.3 Flow diagram of carbonized eggshell powder production

3.3. Calcination of Eggshell Powders

The purpose of calcination of eggshell was to increase the calcium carbonate ($CaCO_3$) content, to reduce eggshell weight, remove protein and membrane from eggshell [50]. The eggshell particle size was reduced and fire in an electric resistance (muffle) furnace at temperature 500 °C for 2 hours [53]. Muffle furnace is ideal for high temperature application up to 1200 ^{0}C for ashing organics, heat treating tools and melting experiments. The calcined eggshell was easy to grind or reduce to a specific particle size and compatible with a wide range of matrix materials.

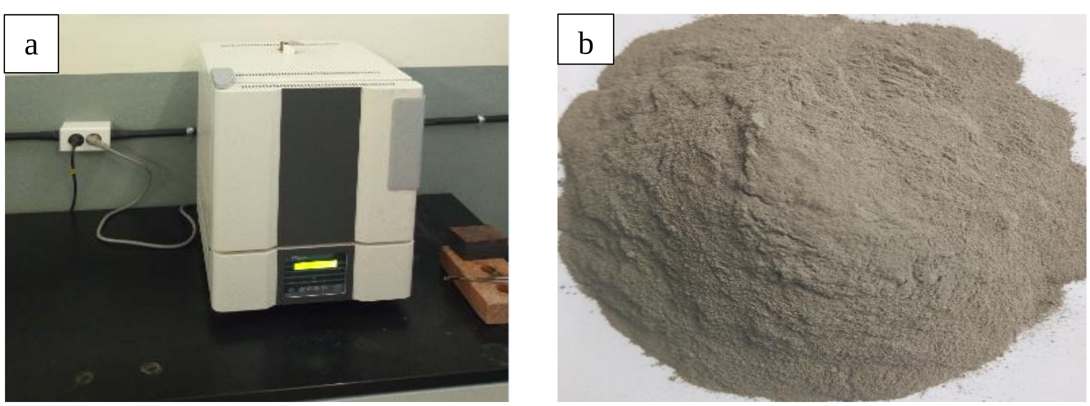

Figure 3.4 (a) Muffle furnace, (b) Carbonized (calcined) eggshell

3.4. Specimens preparation

In this work, 68 specimens for tensile strength test (54 specimens for control parameters optimization process, 8 specimens for confirmation test and 6 specimens for aluminum (AA6063) alloy)/eggshell composite test at optimum condition and 34 specimens for hardness test (27 specimens for control parameters optimization process, 4 specimens for confirmation test, 3 specimen for aluminum (AA6063) alloy/eggshell composite test at optimum condition and 4 specimens for microstructure study) were prepared according to their standards. The specimen was prepared by considering various process parameters and all the steps for the preparation of aluminum (AA6063) alloy/eggshell composites were followed.

3.5. Fabrication Process of AA6063 Alloy/Eggshell Composite

The stir casting procedure was the least complex and economically utilized system. This study was planned to fabricate and investigate the effect "eggshell" powders on the composition material prepared. These methods used to obtain uniform distribution of the eggshell powders in the molten metal to acquired eggshell/AA6063 alloy composite with ideal properties. The required amount of AA6063 alloy was kept in an electric furnace and subsequently the temperature of the furnace is increased to 750℃ [94]. The composite specimens were prepared for a volume fractions of 0%, 5%, 10% and 15% by weight of calcined eggshell added to aluminum (AA6063) alloy [93]. During the addition of eggshell, the mixture was continuously stirred at 200 rpm to obtain ideal composite material. Drilling machine was used to rotate the stirrer at 200 rpm. The molten metal was continuously stirred for 10 minutes in order to ensure a near-uniform distribution of alloying elements. The mixture was then poured into sand molds and allow to solidify to obtain the required specimens. Figure 3.5 shows the stir casting procedure followed to fabricate different composition specimens.

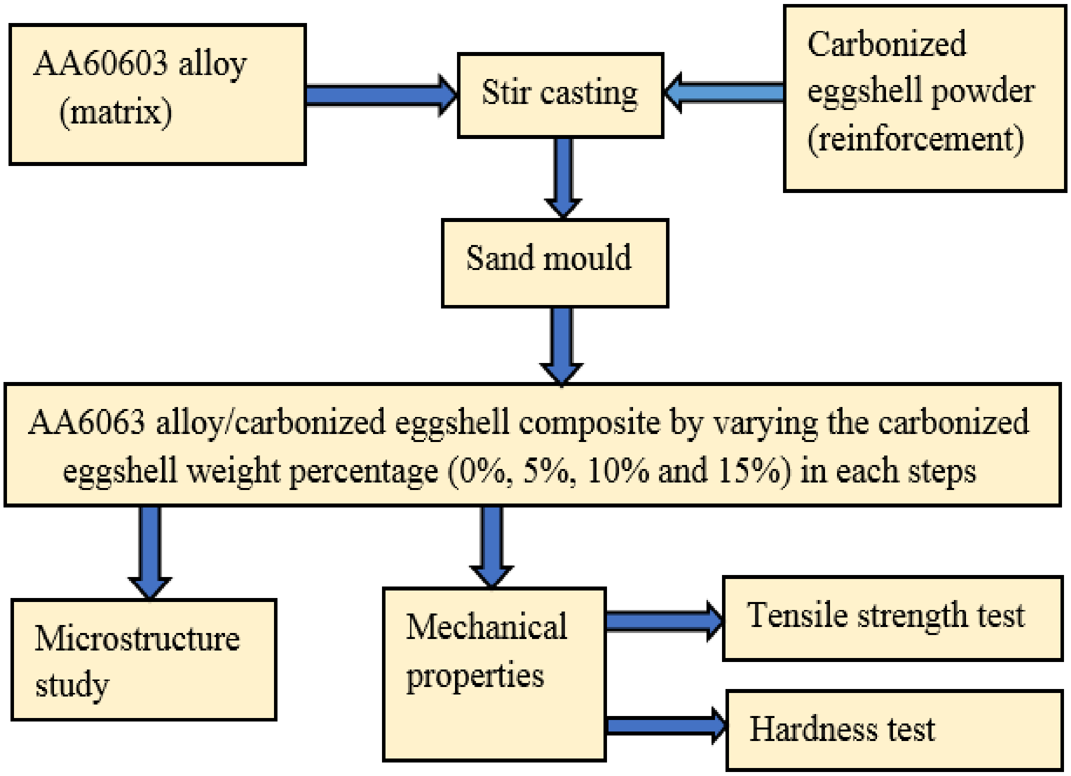

Figure 3.5 Aluminum (AA6063) alloy/eggshell composite process flow diagram

Figure 3.6 presents the 3D section view of stir casting setup used to melt aluminum (AA6063) alloy to obtain composite material.

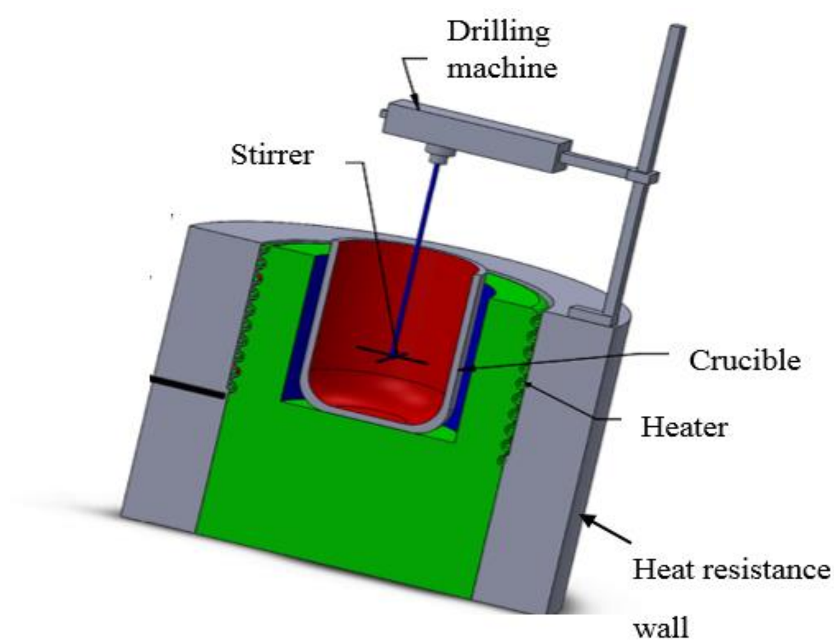

Figure 3.6 3D section view of stir casting setup.

There were several process parameters that influence the final microstructure and mechanical properties (hardness and tensile strength) of the composites were eggshell weight %, stirring speed, stirring temperature, stirring time, preheated temperature of reinforcement, pouring temperature, mold temperature and etc.

3.5.1. Sand Mold Casting

Casting was a metal object produced by allowing molten metal to solidify in a mold. The shape of the object was determined by the shape of the mold cavity. The mold cavity cleaned before pouring the liquid metal, so that it reduced the solidification rate. The liquid metal matrix composite was taken out and poured in to the mold cavity then cooled. After casting, the specimens which had circular shape were prepared by machining for microstructural studies, tensile and hardness testing for the purpose of determining its mechanical properties. Figure 3.7 show the 3D half-section view of sand mold used for making microstructure study, tensile and hardness test specimens.

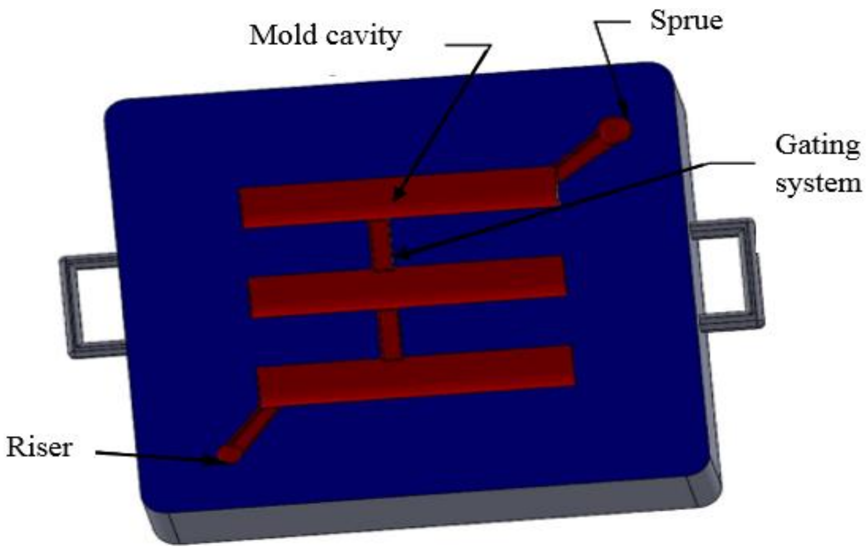

Figure 3.7 3D section view of sand mold

3.5.2. Machining of Tensile Test Specimens

The prepared cylindrical specimens were brought down to final dimensions (gauge diameter 8mm and length 125mm) as per ASTM E8 Standard by machining the specimens using conventional lathe machine. The machined specimens were proposed for tensile strength test. Figure 3.8 provides the dimension of tensile test specimen.

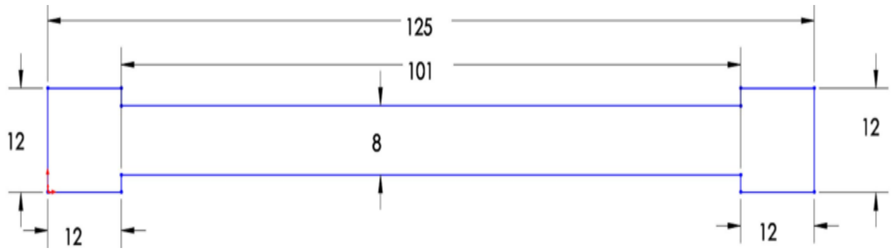

Figure 3.8 Dimensions of tensile test specimen

3.5.3. Machining of Hardness Test Specimens

The prepared cylindrical specimens were brought down to final dimensions (diameter 20mm and length 25mm) as per ASTM E 384 standard by machining the specimen using conventional lathe machine. Figure 3.9 provides the dimension of hardness test specimen.

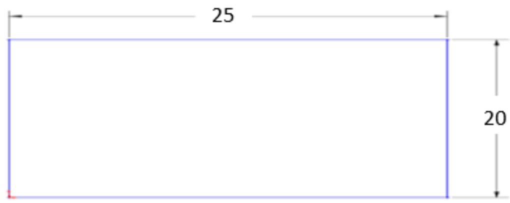

Figure 3.9 Dimensions of hardness test specimen

3.6. Tests for Tensile Strength, Hardness and Microstructure Specimens

3.6.1. Tensile Test Specimens

Tensile strength test was carried out on the prepared composite specimens using a computerized universal testing machine as per the ASTM E8 standard. The Tensile load was applied gradually from initial value of 0 KN till failure load. Two specimens were tested for each composition for the tensile strength. The specimens used for tensile test were shown in figure 3.10.

Figure 3.10 Tensile test specimens

3.6.2. Hardness Test Specimens

Hardness test was carried out at room temperature using ASTM E 384 standard Vickers hardness tester with at two indentations of each specimen and then the average values were utilized to calculate hardness number. The Specimens were grinded and polished with 400, 800, and 1200 grit emery paper followed by dry and wet polishing in polishing machine to get fine surface before testing their hardness. The hardness test is conducted through Vickers hardness tester having a microscope of magnification 125X and 250X. Load used on Vickers hardness tester 98.07 Kgf (10N) at dwell time of 15 seconds for each specimen. The diagonal length was more closely measured, taking measurements between 0.3-0.5 mm. After the diagonal length measurement, the values of the Vickers hardness were calculated by the following equation:

$$VH = \frac{0.102 * 2F[\sin\frac{136^{o}}{2}]}{d^2} \qquad (3.1)$$

where VH is the Vickers hardness of composite specimens; F is the applied load (kgf) and d is the arithmetic mean of the two diagonals, $d_1$and d_2 (mm). Specimens used for hardness test were shown in figure 3.11.

Figure 3.11 Hardness test specimens

3.6.3. Microstructure test specimens

The microstructure specimens were prepared with the dimensions of diameter 20 mm and length 5 mm using abrasive cutter machine. The specimens were prepared for microstructural study as per ASTM E3-11 standard. The cross-sections of these specimens were ground with 400–1200 grit silicon carbide papers, polished using diamond suspension. The polished specimens were cleaned with acetone and etched with (Methanol (25ml) + Hydrochloric (25ml) + Nitric Acid (25ml) + Hydrofluoric acid 1 drop) [70]. The microstructure of these specimens was observed using optical microscope (model: Huvitz HR-300 series and magnification x1000). Microstructure specimens used for microstructure study were shown in figure 3.12.

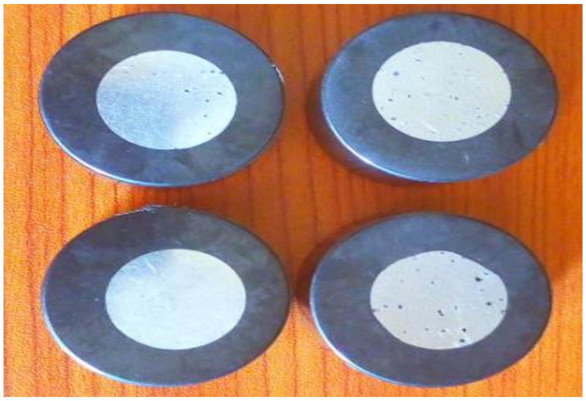

Figure 3.12 Microstructure test specimens

3.7. Optimization procedure for the Stir Casting Process

The Taguchi method involves reducing the variation in a process through robust design of experiments. The overall objective of the method is to produce high quality product at low cost to the manufacturer. The Taguchi method was developed by Genichi Taguchi. He developed a method for designing experiments to investigate how different parameters affect the mean and variance of a process performance characteristic that defines how well the process is functioning. The experimental design proposed by Taguchi involves using orthogonal arrays to organize the parameters affecting the process and the levels at which they should be varied. Instead of having to test all possible combinations like the factorial design, the Taguchi method tests pairs of combinations. This allows for the collection of the necessary data to determine which factors most affect the product quality with a minimum amount of experimentation, thus saving time and resources. The Taguchi method is best used when there is an intermediate number of a variable, few interactions between variables, and when only a few variables contribute significantly.

3.8. Procedures of Taguchi's Method

Taguchi approach to parameter design are provides the design engineer with a systematic and efficient method for determining optimum design parameters for performance and. The objective is to select the best combination of control parameters so that the product or process is most robust with respect to noise factors. Taguchi approach a standard step procedure for applying of method for optimizing any process parameter design using the orthogonal array experimental design proposed by Taguchi (DOE). DOE provides a powerful means to achieve important improvements in product quality and process efficiency. The performance parameter (output) is noted for each experimental run for analysis.

The design of experiment by means of Taguchi approach is selected to find the optimum parameters among the effective factors by running a number of experiments. The process of performing a Taguchi experiment could be summarized in distinct steps:

- Define the process objective.
- Identify the noise factors.
- Identify control factors and their levels.
- Choose orthogonal array as per number of factors and level.
- Conduct experiments as per orthogonal array and observe the change in critical properties.
- Data Analysis-S/N Ratio.
- Determination of optimum setting parameters.
- Confirmation experiment to check whether there is improvement.

3.8.1. Selection of the process objective

The measured response for the process output were chosen to be tensile strength and hardness. This thesis objective is to find the best value of tensile strength and hardness of aluminum (AA6063) alloy/eggshell composite. The signal-to-noise (S/N) ratio is calculated for each factor level combination. larger-the-better is used where the designer wants to maximize the system response since quality increases with the system response.

$$S/N = -10 log \left[\frac{1}{r}\sum_{i=1}^{1}\left(\frac{1}{y_i}\right)^2\right] \tag{3.2}$$

Here db. means decibel, y = observation, r = total number of observation and y_i = ith response

3.8.2. Stir Casting Process Parameters

There are two types of factors that affect a product’s functional characteristic: control factors and noise factors.

Control Factors: is also an input to the system. Values/level is fixed at the optimum level for the best performance. Control factors are those factors which can easily be controlled such as eggshell weight percentage, stirring temperature, stirring time and stirring speed in stir casting process.

Noise Factors: is an uncontrollable factor and difficult or impossible or too expensive to control or the undesirable and uncontrollable sources that can cause deviation from better values in product’s functional characteristics are called noise. There are three types of noise factors: outer noise, inner noise, and between product noise.

External Noise: Operating environment variation such as outside temperature and humidity, and conditions of use that disturb the functions of a product.

Internal Noise: These are internal to the product or process.

Unit-to-unit Noise: The differences between individual products because of manufacturing process imperfections such as variations in machine setting (http://www.worldscibooks.com). Noise factors are primarily response for causing a product's performance to deviate from its better value. The environment in which experiments are performed is the main external source of variation of performance of stir casting process. Each type of noise factor and controllable factors in product and process design are show in Figure 3.13.

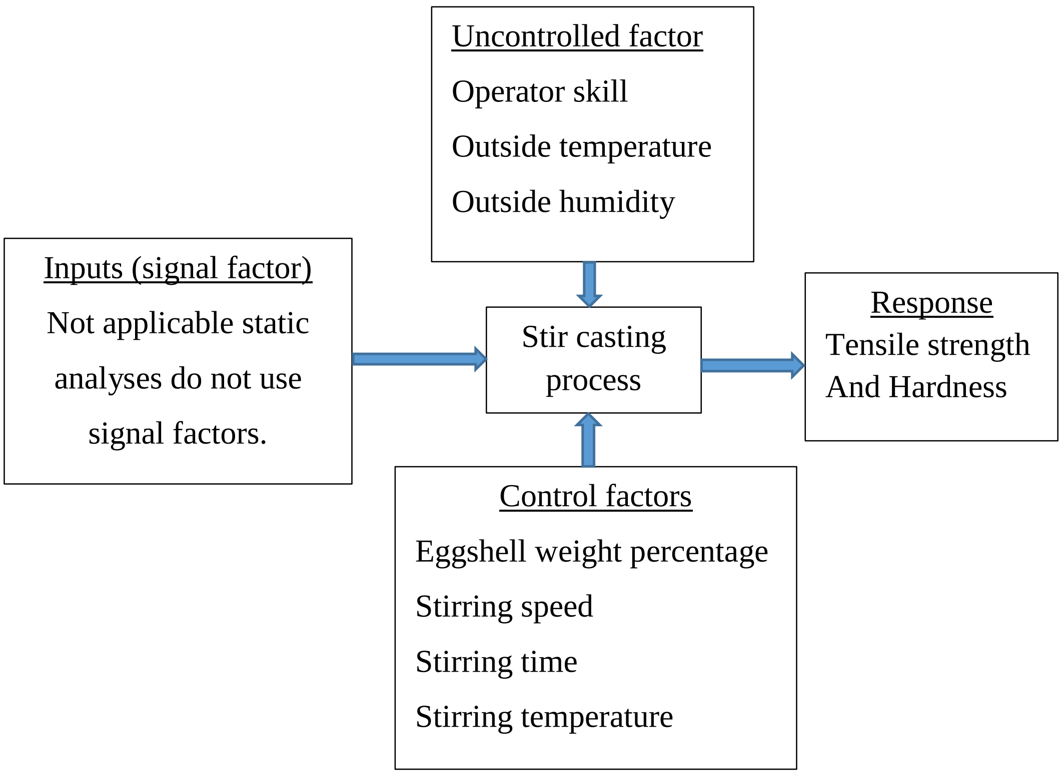

Figure 3.13 Parameters of stir casting process

3.8.3. Design of Experiment

Design of Experiments technique has been utilized to obtain the best combination of design factors to achieve better tensile strength and hardness uniformity on specific aluminum (AA6063) alloy/eggshell composite. Stir casting involves several input parameters to be considered during fabrication process. In this thesis, the combination factors such as eggshell weight percentage, stirrer speed, stirring time and stirring temperature are considered. These factors are the most important to have the best tensile strength and hardness value, the design variables can be summarized as follows:

- ❖ Three levels of the eggshell weight percentage from 5 wt.%-15 wt.%
- ❖ Three levels of the stirrer speed from 200rpm-400rpm
- ❖ Three levels of stirring times 5sec-15sec
- ❖ Three levels of the stirring temperatures from 700℃-800℃

For conducting the experiments, it has been decided to follow the Taguchi method of experimental design and an appropriate orthogonal array is to be selected after taking into consideration the above design variables. Out of the above listed design variables, the orthogonal array was to be selected for four design variables (namely, eggshell particle sizes, stirrer speeds, stirring times and stirring temperatures) which would constitute the L27 orthogonal array.

The most important outputs are tensile strength and hardness values, the same have been selected as response parameters for this research work also. The experimental data will be analyzed as per Taguchi method to find out the optimum stir casting process condition and percentage contribution of each factor. The following stir casting process parameters were kept fixed.

- ❖ **Fixed Parameters**
  - Eggshell particle size
  - Blade design of stirrer
  - Mold Temperature
  - Type of material
  - Powder feed rate
  - The gating design
  - The mold condition

3.8.4. Selection of orthogonal array

In this experiment, there are four parameters at three levels each. The degrees of freedom for the factors under investigation, D, assuming no interactions is given as:

$$D = F(L-1)$$

Where, D is degree of freedom, F number of factors and L is number of level for each factor.

The total degree of freedom available in an OA, Do, is equal to the number of run (N) minus 1.

$$D=N-1$$

In order to select the particular orthogonal array for an experiment the following inequality must be satisfied

$$D_O \geq D$$

The DOF of the orthogonal array selected should have higher than that of total DOF of the experiment. Table 3.4 presents Stir casting parameters and their levels.

Table 3.4: Stir casting parameters and their levels.

Parameter	Unit	Level 1	Level 2	Level 3	DOF
Eggshell weight percentage	gm	5	10	15	2
Stirrer speed (B)	rpm	200	300	400	2
Stirring times (C)	$sec.$	5	10	15	2
Stirring temperature (D)	℃	700	750	800	2

This experiment has four variables (factors) at three levels full factorial experiment would require 3^4=81 experiments test to variables. But using Taguchi's orthogonal arrays, only 27 experiments runs are necessary. In this study Taguchi experiment with a L27 (3^4) orthogonal array (27 tests, 4 factors and 3 levels) were used. Table 3.5 columns represent the control factors, the table rows represent the runs (combination of factor levels), and each table cell represents the factor level for that run.

Table 3.5: Experimental layout in coded factor levels

Run	Eggshell weight percentage ($wt.\%$)	Stirrer speed (rpm)	Stirring times ($sec.$)	Stirring temperature (℃)	Designation			
					A	B	C	D
1	5	200	5	700	1	1	1	1
2	5	200	5	700	1	1	1	1
3	5	200	5	700	1	1	1	1
4	5	300	10	750	1	2	2	2
5	5	300	10	750	1	2	2	2
6	5	300	10	750	1	2	2	2
7	5	400	15	800	1	3	3	3
8	5	400	15	800	1	3	3	3
9	5	400	15	800	1	3	3	3
10	10	200	10	800	2	1	2	3
11	10	200	10	800	2	1	2	3
12	10	200	10	800	2	1	2	3
13	10	300	15	700	2	2	3	1
14	10	300	15	700	2	2	3	1
15	10	300	15	700	2	2	3	1
16	10	400	5	750	2	3	1	2
17	10	400	5	750	2	3	1	2
18	10	400	5	750	2	3	1	2
19	15	200	15	750	3	1	3	2
20	15	200	15	750	3	1	3	2
21	15	200	15	750	3	1	3	2
22	15	300	5	800	3	2	1	3
23	15	300	5	800	3	2	1	3
24	15	300	5	800	3	2	1	3
25	15	400	10	700	3	3	2	1
26	15	400	10	700	3	3	2	1
27	15	400	10	700	3	3	2	1

CHAPTER 4: ANALYSIS AND INTERPRETATION METHODS

4.1. Experimental Analysis and Results

Table 4.1 show measured Universal tensile strength and Vickers hardness experimental values of the aluminum (AA6063) alloy/eggshell composite specimen for each trial. From the experimental results, experiments have the highest and the lowest values for both tensile strength and hardness values.

Table 4.1: Measured experimental results for tensile strength and hardness

Run	A	B	C	D	Tensile strength		Hardness	
					Trial 1	Trial 2	T1	T2
1	5	200	5	700	120.14	128.67	96.34	101.6
2	5	200	5	700	120.09	126.41	99.01	95.21
3	5	200	5	700	122.13	129.22	91.45	96.69
4	5	300	10	750	137.11	135.90	102.24	99.56
5	5	300	10	750	133.02	138.46	109.54	98.42
6	5	300	10	750	130.00	133.44	99.67	98.71
7	5	400	15	800	118.22	117.77	86.66	91.34
8	5	400	15	800	116.43	114.32	93.83	89.13
9	5	400	15	800	114.72	115.12	87.12	86.34
10	10	200	10	800	153.03	158.43	114.47	108.36
11	10	200	10	800	166.66	162.47	117.57	109.45
12	10	200	10	800	149.06	155.03	114.90	116.90
13	10	300	15	700	133.82	126.48	98.43	102.03
14	10	300	15	700	128.65	126.44	94.61	97.45
15	10	300	15	700	123.03	127.30	91.56	93.45
16	10	400	5	750	144.24	146.23	103.51	111.23
17	10	400	5	750	138.06	153.68	108.89	106.67
18	10	400	5	750	141.86	149.34	107.22	101.10
19	15	200	15	750	126.43	123.56	89.34	83.78
20	15	200	15	750	132.92	121.59	82.67	83.51
21	15	200	15	750	129.07	127.44	75.52	87.54
22	15	300	5	800	118.16	116.49	77.17	76.67
23	15	300	5	800	119.82	115.50	76.61	79.45
24	15	300	5	800	114.20	114.95	72.23	71.59
25	15	400	10	700	129.92	124.98	91.23	88.45
26	15	400	10	700	124.65	123.66	82.34	83.66
27	15	400	10	700	130.48	128.52	89.78	87.56

4.2. Analysis of Mean for Tensile Strength and Hardness

The average main value of tensile strength for each factor i.e. A, B, C and D calculated using the Minitab 17 design of experiments. Table 4.2 presents the experimental results of Universal tensile strength and Vickers hardness test for each specimen and their mean values. In this study each Universal tensile strength and Vickers hardness trial value is the average of two trial reading.

Table 4.2: Mean values for tensile strength and hardness results

Run	A	B	C	D	Tensile strength			Hardness		
					Trial 1	Trial 2	Mean	T1	T2	Mean
1	5	200	5	700	120.14	128.67	124.405	96.34	101.6	98.97
2	5	200	5	700	120.09	126.41	123.25	99.01	95.21	97.11
3	5	200	5	700	122.13	129.22	125.675	91.45	96.69	94.07
4	5	300	10	750	137.11	135.90	136.505	102.24	99.56	100.90
5	5	300	10	750	133.02	138.46	135.74	109.54	98.42	103.98
6	5	300	10	750	130.00	133.44	131.72	99.67	98.71	99.19
7	5	400	15	800	118.22	117.77	117.995	86.66	91.34	89
8	5	400	15	800	116.43	114.32	115.375	93.83	89.13	91.48
9	5	400	15	800	114.72	115.12	114.92	87.12	86.34	86.73
10	10	200	10	800	153.03	158.43	155.73	114.47	108.36	111.415
11	10	200	10	800	166.66	162.47	164.565	117.57	109.45	113.51
12	10	200	10	800	149.06	155.03	152.045	114.90	116.90	115.9
13	10	300	15	700	133.82	126.48	130.15	98.43	102.03	100.23
14	10	300	15	700	128.65	126.44	127.545	94.61	97.45	96.03
15	10	300	15	700	123.03	127.30	125.165	91.56	93.45	92.505
16	10	400	5	750	144.24	146.23	145.235	103.51	111.23	107.37
17	10	400	5	750	138.06	153.68	145.87	108.89	106.67	107.78
18	10	400	5	750	141.86	149.34	145.6	107.22	101.10	104.16
19	15	200	15	750	126.43	123.56	124.995	89.34	83.78	86.56
20	15	200	15	750	132.92	121.59	127.255	82.67	83.51	83.09
21	15	200	15	750	129.07	127.44	128.255	75.52	87.54	86.53
22	15	300	5	800	118.16	116.49	117.325	77.17	76.67	76.92
23	15	300	5	800	119.82	115.50	117.66	76.61	79.45	78.03
24	15	300	5	800	114.20	114.95	114.575	72.23	71.59	71.91
25	15	400	10	700	129.92	124.98	127.45	91.23	88.45	89.84
26	15	400	10	700	124.65	123.66	124.155	82.34	83.66	83
27	15	400	10	700	130.48	128.52	129.500	89.78	87.56	88.67
Average performance mean value (m)							130.691			94.625

The mean value of tensile strength for each factor i.e. A, B, C, and D at each level i.e. level 1, level 2 and level 3 are obtained and the result is listed in response table 4.3.

Table 4.3: Tensile strength response table for mean

Symbols	Parameters	Means level 1	Means level 2	Means level 3	Delta	Rank
A	Eggshell particle sizes	125.65	**143.545**	123.463	**20.082**	1
B	Stirrer speed	**136.242**	126.265	**129.567**	9.977	3
C	Stirring time	128.844	139.712	123.517	16.195	2
D	Stirring temperature	126.366	**135.686**	130.021	9.32	4

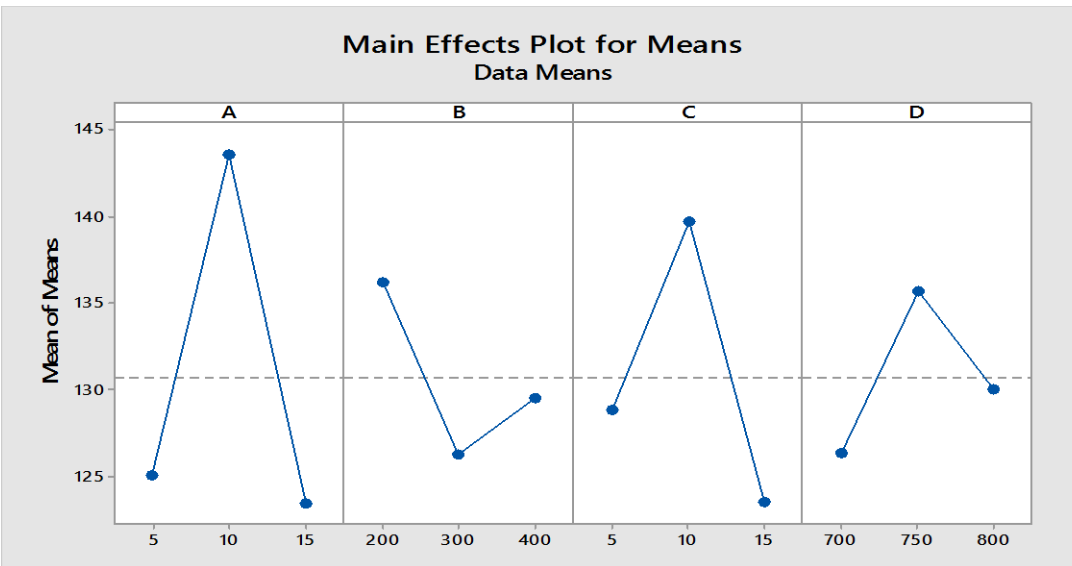

Figure 4.1 Tensile strength main effects plot for means

The control factors main effect plot for means tensile strength variations is shown in the figure 4.1.

Delta = (maximum mean – minimum mean)

Table 4.3 and Figure 4.1 show that the factor with the greatest effect on the mean is eggshell weight percentage (Delta = 20.082, Rank = 1). The factor with the least effect on the mean is stirring temperature (Delta = 9.32, Rank = 4). Since main effect plots for means is helpful for adjusting the mean on better value, those parameter levels with better tensile strength of specimens that means $\mathbf{A_2B_1C_2D_2}$ will be taken as a possible parameter combination for better values.

The average main value of hardness for each factor i.e. A, B, C, and D at each level i.e. level 1, level 2 and level 3 are obtained and the result is listed in response table 4.4.

Table 4.4: Hardness response table for mean

		Means			Delta	Rank
Symbols	Parameters	level				
		1	2	3		
A	Eggshell weight percentage	95.714	**105.433**	82.728	22.705	1
B	Stirrer speed	**98.573**	91.077	94.226	7.496	3
C	Stirring time	92.924	**100.712**	90.239	10.473	2
D	Stirring temperature	93.381	**97.729**	92.766	4.963	4

Figure 4.2 Hardness main effects plot for means

The hardness response table and main effects plot for means both show that the factor with the greatest effect on the mean is eggshell weight percentage (Delta = 22.705, Rank = 1). The factor with the least effect on the mean is stirring temperature (Delta = 4.963, Rank = 4). The parameters levels with better hardness values will be taken as a possible parameter combination ($A_2B_1C_2D_2$) for better values.

4.2.1. Predict the Performance at Optimal Levels the mean value

For predicting the optimum response approximately, four factors of parameters are selected by mean value. $A_1B_1C_2D_3$ is the optimum levels of parameters mean value for both tensile strength and hardness. To find out predicted value of the responses (tensile strength and hardness) the following equation is used:

$$Y_{opt} = m + (m_{A_2} - m) + (m_{B_1} - m) + (m_{C_2} - m) + (m_{D_2} - m) \tag{4.1}$$

Where, m = Average performance mean value for both tensile strength and hardness

m_{A_2} = optimum level value for parameter A

m_{B_1} = optimum level value for parameter B

m_{C_2} = optimum level value for parameter C

m_{D_2} = optimum level value for parameter D

Y_{opt} = 130.691 + (143.545 – 130.691) + (136.242 – 130.691) + (139.712 – 130.691) + (135.686 – 130.691) = 158.117 (predict mean value for tensile strength)

= 94.625 + (105.433 – 94.625) + (98.573 – 94.625) + (100.712 – 94.625) + (97.729 – 94.626) = 118.572 (predict mean value for hardness)

4.3. Analysis of S/N Ratio for Tensile Strength and Hardness

The Taguchi method uses S/N ratio to measure the variations of the tensile strength and hardness experimental design. The equations for calculating the signal to noise ratios are based on the characteristics of the response variables being evaluated; nominal the best, smaller the better and larger the better. In the case of this study, the response variables are large tensile strength and hardness. Therefore, larger the better characteristic was used and the S/N equation for this characteristic is given as:

$$\text{S/N} = -10log\left[\frac{1}{r}\sum_{i=1}^{1}\left(\frac{1}{y_i}\right)^2\right] \tag{4.2}$$

Here db. means decibel, y = observation, r = total number of observation and y_i = ith response

Table 4.5 presents the calculations of S/N Ratios for tensile strength and hardness results using the Minitab 17 design of experiments.

Table 4.5: Analysis of S/N Ratios for tensile strength and hardness

Run	A	B	C	D	Tensile strength		Hardness	
					Mean	S/N	Mean	S/N
1	5	200	5	700	124.405	41.896	98.97	39.910
2	5	200	5	700	123.25	41.816	97.11	39.745
3	5	200	5	700	125.675	41.985	94.07	39.469
4	5	300	10	750	136.505	42.703	100.90	40.078
5	5	300	10	750	135.74	42.654	103.98	40.339
6	5	300	10	750	131.72	42.393	99.19	39.929
7	5	400	15	800	117.995	41.437	89	38.988
8	5	400	15	800	115.375	41.242	91.48	39.226
9	5	400	15	800	114.92	41.208	86.73	38.763
10	10	200	10	800	155.73	43.847	111.415	40.939
11	10	200	10	800	164.565	44.327	113.51	41.101
12	10	200	10	800	152.045	43.639	115.9	41.282
13	10	300	15	700	130.15	42.289	100.23	40.020
14	10	300	15	700	127.545	42.113	96.03	39.648
15	10	300	15	700	125.165	41.950	92.505	39.323
16	10	400	5	750	145.235	43.241	107.37	40.618
17	10	400	5	750	145.87	43.279	107.78	40.651
18	10	400	5	750	145.6	43.263	104.16	40.354
19	15	200	15	750	124.995	41.938	86.56	38.746
20	15	200	15	750	127.255	42.094	83.09	38.391
21	15	200	15	750	128.255	42.161	86.53	38.743
22	15	300	5	800	117.325	41.388	76.92	37.721
23	15	300	5	800	117.66	41.413	78.03	37.845
24	15	300	5	800	114.575	41.182	71.91	37.136
25	15	400	10	700	127.45	42.107	89.84	39.069
26	15	400	10	700	124.155	41.879	83	38.382
27	15	400	10	700	129.500	42.345	88.67	38.956
Average performance S/N results						42.285		39.458

After calculating the S/N ratio for each experiment, the average S/N value is calculated for each factor and level as shown in Table 4.6.

Table 4.6: Tensile strength response table for S/N ratio

Symbols	Parameters	Signal to Noise ratios level 1	2	3	Delta	Rank
A	Eggshell weight percentage	41.925	**43.102**	41.821	1.281	1
B	Stirrer speed	**42.630**	42.008	42.210	0.622	3
C	Stirring time	42.162	**42.861**	41.824	1.037	2
D	Stirring temperature	42.029	**42.635**	42.183	0.336	4

Delta = (maximum S/N ratio – minimum S/N ratio)

By Looking at the tensile strength table and main effects plot for the signal-to-noise (S/N) ratios the eggshell weight percentage have the greatest effect (Delta = 1.281, Rank = 1). The factor with the least effect on S/N ratio is stirring temperature (Delta= 0.336, Rank = 4). Table 4.4 shows that optimal parameter combination on larger is the better quality character is $\mathbf{A_2B_1C_2D_2}$ are the possible parameter combinations for increasing the tensile strength of AA6063 alloy/eggshell composite specimens. The control factors main effect plot for the tensile strength S/N ratio is shown in the figures 4.3.

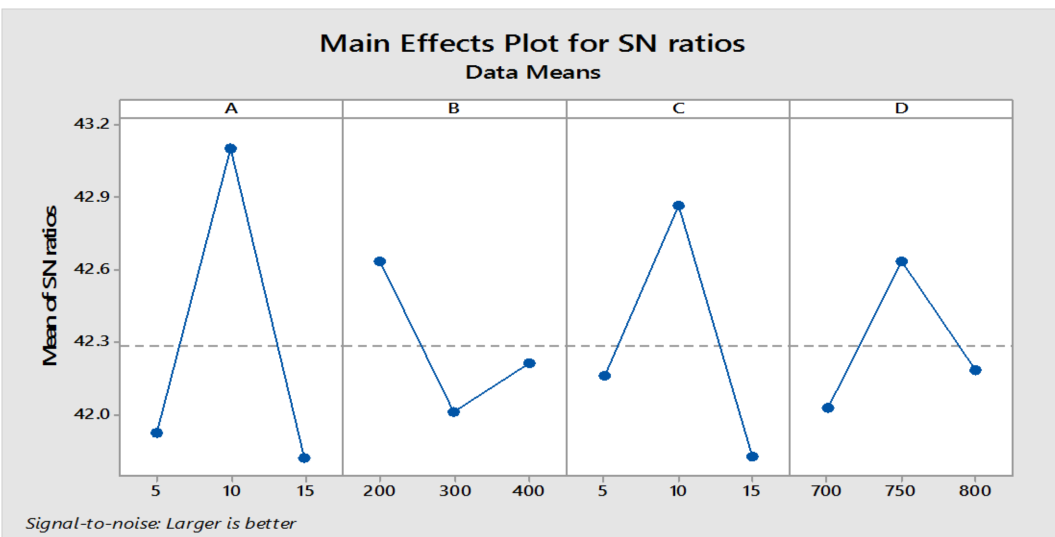

Figure 4.3 Tensile strength main effects plot for S/N ratio

The numerical value of the maximum point in each graph shows the best value of that particular parameter. They also indicate the optimum conditions in the range of the experimental conditions. The most effective parameter to enhance the tensile strength is the eggshell weight percentage. Figure 4.6 shows that the design parameter combination $\mathbf{A_2B_1C_2D_2}$, and the corresponding levels values of each factor for the maximum tensile strength.

Table 4.7: Hardness response table for S/N ratio

Symbols	Parameters	Means level 1	2	3	Delta	Rank
A	Eggshell weight percentage	39.602	**40.433**	38.324	2.109	1
B	Stirrer speed	**39.811**	39.108	39.440	0.703	3
C	Stirring time	39.266	**40.003**	39.089	0.914	2
D	Stirring temperature	39.384	**39.758**	39.217	0.541	4

Table 4.4 shows that optimal parameter combination on larger is the better quality character is $\mathbf{A_2B_1C_2D_2}$ are the possible parameter combinations for increasing the hardness of AA6063 alloy/eggshell composite specimens. By Looking at the hardness tables and main effects plots for the S/N ratios the eggshell weight percentage have the greatest effect (Delta = 2.109, Rank = 1). The factor with the least effect on S/N ratio is stirring temperature (Delta= 0.541, Rank = 4).

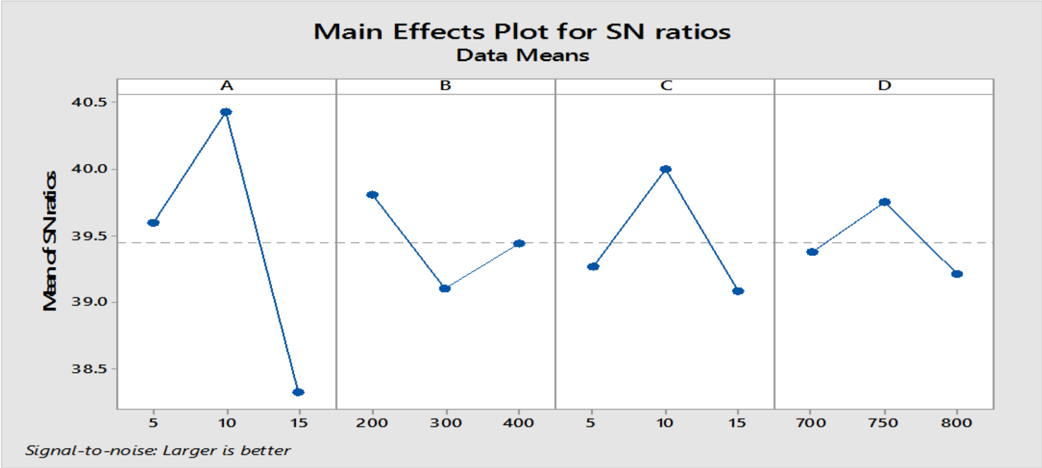

Figure 4.4 Hardness main effects plot for S/N ratio

Figure 4.4 shows that the design parameter combination **$A_2B_1C_2D_2$**, and the corresponding values of each factor for the maximum hardness. The numerical value of the maximum point in each graph shows the best value of that particular parameter. They also indicate the optimum conditions in the range of the experimental conditions. The most effective parameter to enhance the hardness is the eggshell weight percentage.

4.3.1. Predict the Performance at Optimal Levels the S/N Result

For predicting the optimum response approximately, four factors of parameters are selected by high S/N ratio value. $A_2B_1C_2D_2$ is the optimum levels of parameters S/N ratio value for both tensile strength and hardness. To find out predicted values of the responses (tensile strength and hardness) equation 4.1 was used:

$$Y_{opt} = m + (m_2 - m) + (m_{B_1} - m) + (m_{C_2} - m) + (m_{D_2} - m)$$

= 42.285 + (43.102 – 42.285) + (42.630 – 42.285) + (42.861 – 42.285) + (42.635 – 42.285)

= 44.373 dB (predict S/N result for tensile strength)

= 39.458 + (40.433 – 39.458) + (39.811 – 39.458) + (40.003 – 39.458) + (39.758 – 39.458)

= 41.631 dB (predict S/N result for hardness)

4.4. Analysis of variance (ANOVA)

The purpose of the analysis of variance (ANOVA) was to find which parameters significantly affected the quality characteristic. ANOVA is the statistical technique used to find the influence of all control parameters. In the analysis of variance, the percentage contributions of each control factor were used to measure the corresponding effects on the performance characteristics. The significance level of 5%, i.e. for 95 % level of confidence was considered in this analysis. ANOVA results indicate whether or not there is statistically significance difference in output characteristics. ANOVA analysis involves when α values is below 0.05 is significant and above 0.05 is not significant. From analysis of variance Percentage contribution of each influential parameter was calculated by the following formula:

$$P(\%) = \left(\frac{SSTR - DOF * MSE}{SST}\right) * 100 \qquad (4.2)$$

Where,

P: Percentage contribution of individual factor,

SSTR (Adj SS): Sum of squares or treatment sum of squares,

DOF: Degree of freedom,

MSE (Adj SS): Mean square Error,

SST: Total sum of squares.

4.4.1. Analysis of Variance (ANOVA) for Tensile Strength

ANOVA results DOF = degree of freedom, SSTR = Sum of squares, MSE = Main square error and F = F- Ratio calculated by using Minitab 17 software were shown in Table 4.8.

Table 4.8: Analysis of Variance for tensile strength

Source of variation	DoF	SSTR	MSE	F	P	Percent contribution	Significance ($\alpha = 0.05$)
A	2	2242.0	1121.02	141.16	0.000	50.12%	Significant
B	2	465.0	232.49	29.27	0.000	10.4%	Significant
C	2	1226.3	613.16	77.21	0.001	27.44%	Significant
D	2	396.9	198.47	24.99	0.002	8.87%	Significant
Error	18	142	7.94			3.17%	
Total	26	4473.2				100%	

Table 4.8 presents tensile strength percentage contribution variation of factors A, B, C and D. It is observed that all the selected factors have statistical and physical significances on the tensile strength value during stir casting of aluminum (AA6063) alloy/eggshell composite at 95% confidence level.

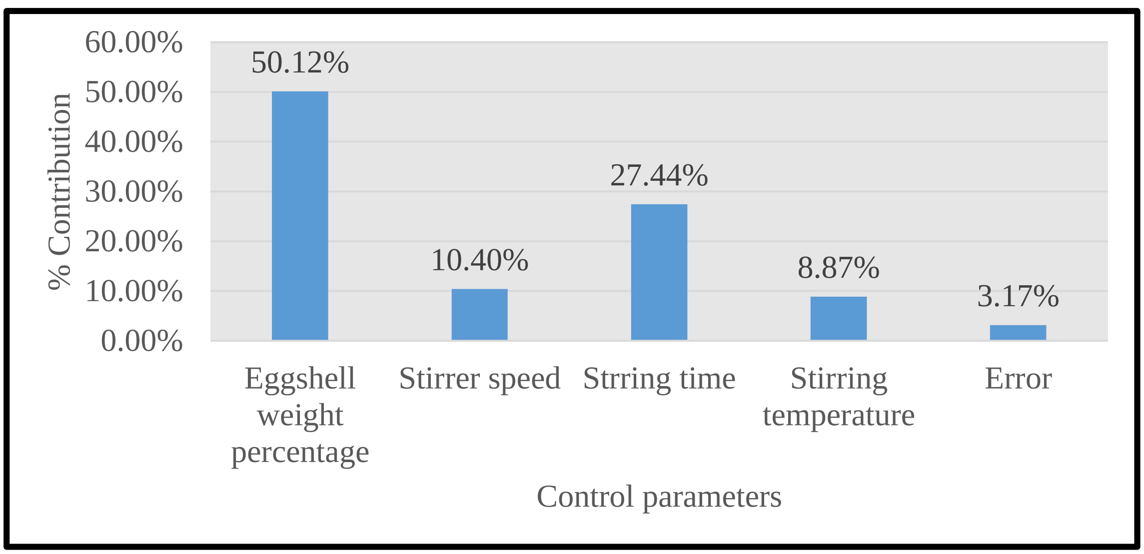

Figure 4.5 Percentage contribution of each control parameter to enhance the tensile strength

Figure 4.5 shows that eggshell weight percentage having 50.12 % contribution and more significant, stirring time having 27.44 % contribution, stirring speed having 10.4 % contribution and stirring temperature having 8.87 % contribution and all these control parameters have significant influence on the tensile strength of AA6063 alloy/eggshell composite specimens.

4.4.2. Analysis of variance (ANOVA) for hardness

Table 4.9 presents hardness percentage contribution variation of factors A, B, C and D.

Table 4.9: Analysis of Variance for hardness

Source of variation	DOF	SSTR	MSE	F	P	Percent contribution	Significance ($\alpha = 0.05$)
A	2	2336.0	1167.98	151.09	0.000	68.82 %	Significant
B	2	255.0	127.49	16.49	0.000	7.51 %	Significant
C	2	532.6	266.28	34.45	0.001	15.69 %	Significant
D	2	131.7	65.87	8.52	0.003	3.88 %	Significant
Error	18	139.1	7.73			4.1 %	
Total	26	3394.4					

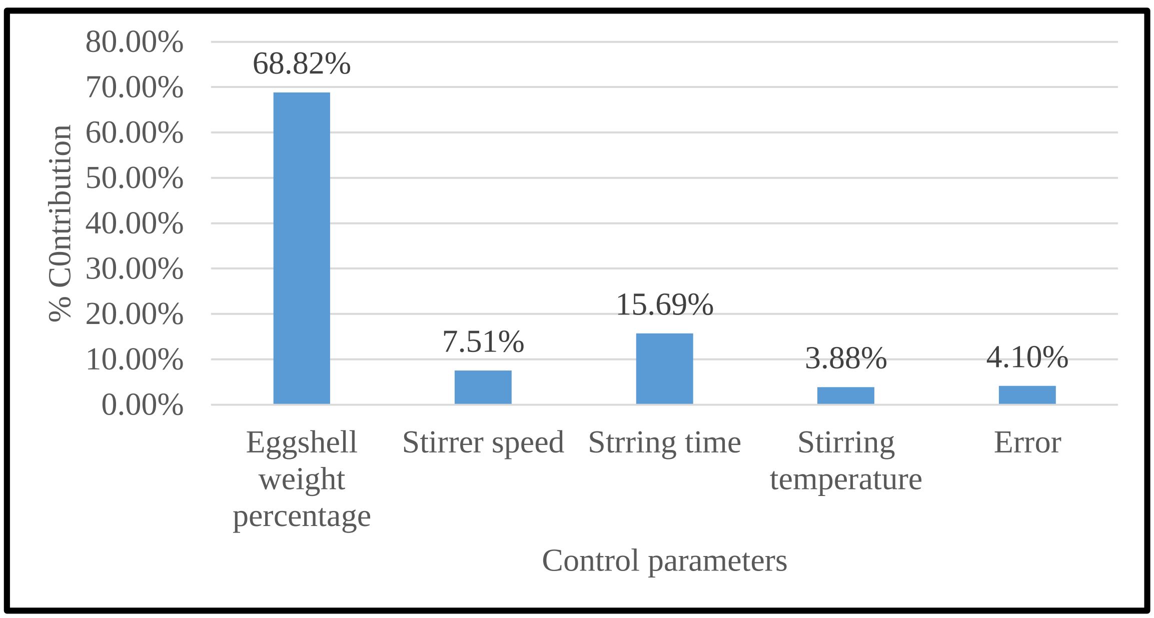

Figure 4.6 Percentage Contribution of Each Control Parameter to Enhance the hardness

From Table 4.9 and Figure 4.6 it is observed that all the selected factors have statistical and physical significances on the hardness value during stir casting of aluminum (AA6063) alloy/eggshell composite at 95% confidence level. From Figure 4.6 it is clearly observed that the eggshell weight percentage (68.82 %), stirrer speed (7.51 %), stirring time (15.69 %) and stirring temperature (3.88 %) have significant influence on the hardness of AA6063 alloy/eggshell composite specimens.

CHAPTER 5: RESULTS AND DISCUSSIONS

5.1. Determining the Optimum Condition

From the Figure 4.5 and 4. 6 it is clearly observed that eggshell weight percentage optimum level value 10, stirrer speed optimum level value 200, stirring time optimum level value 10 and stirring temperature optimum level value 750 make the response S/N ratio values large. Therefore, the optimum processing parameters signal to noise (S/N) ratio is selected. Table 4.10 presents the optimum parameters setting selected for both tensile strength and hardness obtained from the design of experiment and experimental results.

Table 5.1: Optimum parameters setting levels and their values

Controllable factors	Level selected	Value of the level	Rank of affecting the response
Eggshell wt.%	2	10	1
Stirrer speed	1	200	3
Stirring time	2	10	2
Stirring temperature	2	750	4

5.2. Confirmation Test

Once the optimal combination of process parameters and their levels was obtained, the final step was to verify the predicted result against experimental result. If the optimal combination of parameters and their levels coincidently match with one of the experiments in the OA, then no confirmation test is required. As per the DOE analysis and the improvement steps of Taguchi approach, confirmation run is optional to check the predictability of the result. By considering this, the optimum parameter results of eight specimens for the tensile strength and four specimens for the hardness are measured and recorded as shown in Table 5.2 and 5.3 respectively.

Table 5.2: Confirmation experiment results for tensile strength specimens

Run	A	B	C	D	Tensile strength (MPa)			
					T1	T2	Mean	S/N
1	10	200	10	750	156.05	152.43	154.24	43.74
2	10	200	10	750	161.21	158.45	159.83	44.07
3	10	200	10	570	159.61	154.38	157.00	43.92
4	10	200	10	750	162.90	159.98	161.44	44.16
Confirmation experiment mean and S/N results							**158.128**	**43.973**

Table 5.3: Confirmation experiment results for hardness specimens

Run	A	B	C	D	Hardness (VH)			
					T1	T2	Mean	S/N
1	10	200	10	750	118.69	113.17	115.93	41.29
2	10	200	10	750	124.54	120.22	122.38	42.47
3	10	200	10	570	117.36	119.73	118.55	41.67
4	10	200	10	750	115.69	122.28	118.99	41.66
Confirmation experiment mean and S/N results							**118.963**	**41.773**

Table 5.4 presents the confirmation experiment and predicted results comparison for both tensile strength and hardness.

Table 5.4: Comparison between predicted and experimental confirmation

	Combination	Tensile strength (MPa)		Hardness (HV)	
		Mean	S/N ratio	Mean	S/N ratio
Predicted	$A_2B_1C_2D_2$	158.117	44.373	118.572	41.631
Experiment	$A_2B_1C_2D_2$	158.128	43.973	118.963	41.773

Confirmation test was required in this case study because the optimum combination of parameters and their levels ($A_2B_1C_2D_2$) did not correspond to any experiment of the orthogonal array. The optimum combination of parameters and their levels ($A_2B_1C_2D_2$) was produced on the same stir casting furnace and from the same materials. The results of confirmation experiment mean and S/N obtained from the experiment were then compared with the predicted mean and S/N results as shown in Table 5.2 for both tensile strength and hardness. It can be seen from this table that the difference between experimental results and the predicted results are very small. The values of predicted by using Taguchi approach is approximately near to the value which is experimentally obtained. These verify that the experimental results of both tensile strength and hardness are strongly correlated with the predicted results.

5.3. Microstructural Study and analysis of AA6063 Alloy/Eggshell composite

Optical micrographs of AA6063 alloy/eggshell composites were studied for microstructure analysis. The distribution was examined by microstructure analysis, tensile strength and hardness distribution. The micrographs of AA6063 alloy/eggshell composites show that the eggshell particles are uniformly distributed in the AA6063 alloy up to 10 wt.% of eggshell particles, with no porosity or defects observed. There was a good distribution of eggshell particles in aluminum (AA6063) alloy as well as very low agglomeration of eggshell particles in the low percentage (less than 10 wt.%) content. The addition of 15 % eggshell weight percentage in AA6063 alloy result in porosity, agglomeration and creates more defects in casting composite.

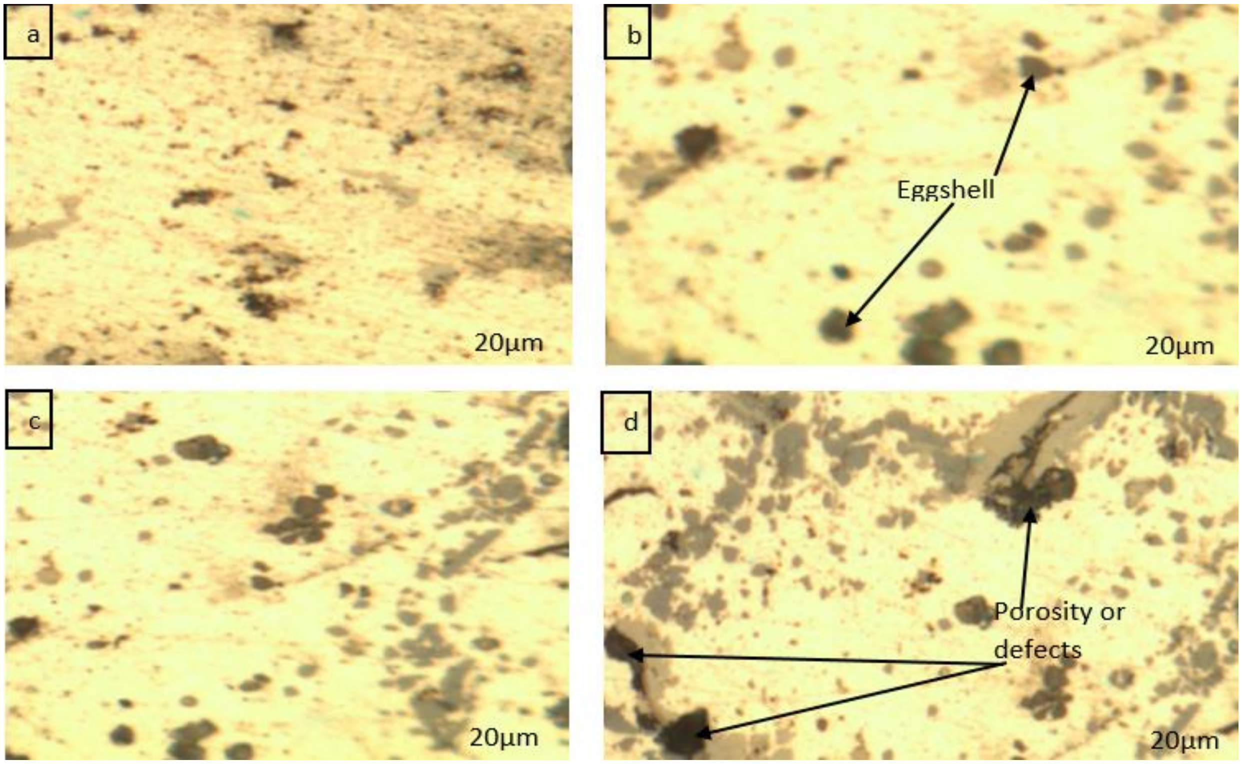

Figure 5.1 Microstructure of AA6063 alloy/eggshell composite with (a) 0 wt.% eggshell, (b) 5 wt.% eggshell, (c) 10 wt.% and (d) 15 wt.% eggshell observed under optical microscope.

Figure 4.1 (a, b, c and d) show the microstructures of the composites with calcined eggshell powder after stir casting process. Figure 4.1 (a) shows that it is pure AA6063 alloy and no eggshell particles distribution obtained in it. In the two cases (5 and 10 eggshell weight percentage), good dispersion of eggshell particle in the aluminum alloy has been observed. There is no large porosity and particles clustering existed in these optical micrographs. The higher percentage eggshell weight (greater than 10 %) leads to a nonhomogeneous eggshell distribution and the formation of eggshell particle clusters. The existence of porosity and eggshell particle clustering was attributed to the high viscosity and low shearing rate of the melt. Viscosity and shearing rate decreased when increasing process (stirring) temperature. This can be attributed more eggshell particles clustering in the AA6063 alloy. When AA6063 alloy/eggshell particles were stirred for a constant speed with longer stirring time at higher temperature, the eggshell particles were agglomerated in the melt. During higher stirring time with higher temperature, the geometry of capturing eggshell particles does not restrict their movement inside the liquid metal as well as solidification. Also the presence of a low viscosity of liquid metal tends to physically not restricts growth of porosity. Thus, the tendency for eggshell particle cluster or porosity is high in the higher temperature with prolonged contact between AA6063 alloy and eggshell powder. Finally, the eggshell particle clustering and agglomeration result in partial ductile and brittle which result in lower mechanical properties of AA6063 alloy/eggshell composite material.

5.4. Distribution of Calcined Eggshell in Aluminum (AA6063) Alloy

The distribution of eggshell powder in aluminum (AA6063) alloy are one of the key factors influencing the properties of the material. When the composite has been developed without applying stirring process, eggshell particle clustering occurred in some places, and some places were identified without eggshell particles inclusion. This was due to the fact that when the eggshell particles were added into the molten AA6063 alloy, it was that observed to be floating on the surface, though they have a small specific density than the molten metal (AA6063 alloy). This was due to high surface tension and poor wetting between the particles and the melt. Therefore, stirring system was applied to overcome surface tension to improve wettability.

To improve the eggshell powder distribution, aluminum (AA6063) alloy was heat above its liquidus temperature and stir the molten metal using drilling machine for 10 minutes at 200 rpm. This causes uniform distribution of eggshell powder with low agglomeration and porosity. When the specimens were prepared at 750˚C, the eggshell particles were distributed uniformly in

AA6063 alloy. The specimens containing 0%, 5%, 10% and 15% eggshell weight are developed by stir casting method.

5.5. Effect of Calcined Eggshell on mechanical properties of AA6063 alloy

The purpose of calcination of eggshell was to increase the calcium carbonate ($CaCO_3$) content, to reduce eggshell weight, remove protein and membrane from eggshell. The eggshell particle size was reduced and fire in an electric resistance furnace at temperature between 200-600 °C for 2 hours. The calcined eggshell was easy to grind or reduce to a specific particle size and compatible with a wide range of matrix materials. Chemically modified chicken eggshell acts as an effective reinforcement that enhances the mechanical properties of aluminum (AA6063) alloy.

5.6. Wettability of Aluminum AA6063 Alloy/Eggshell composite

The distribution of eggshell powder into the AA6063 alloy plays a significant role on the mechanical properties of the composite materials. Optical microscope can be used to evaluate the eggshell powders dispersion in the composite. In order to achieve high level of mechanical properties in the composite, a good inter facial bonding (wettability) between the eggshell powders and the liquid AA6063 alloy has to be investigated.

The gas layers are also the main factor for poor wettability. As the amount of added eggshell powder increases (beyond 10 weight percentage), the amount of trapped air increases there by increasing the amount of porosity resulting in mechanical properties decrease. Firstly, gas layers can cause migration of particles, making it difficult to incorporate the particles into the melt. Secondly, even the particles can be suspended in the melt by vigorous stirring; it has been still difficult for the particles to be wetted by the molten metals because of the gas layers. From the above analysis it was necessary to break the gas layers by considering optimum values of process parameters such as stirring speed, stirring time and stirring temperature in order to achieve good wettability. The effect of stirring time in the AA6063 alloy/eggshell composites mainly two ways: to distribute the particles in the liquid, and to create perfect interface bond between eggshell and AA6063 alloy (wettability).

5.7. Density of AA6063 Alloy/Eggshell composite specimens

The experimental tests were performed on four specimens, for every weight percentage of eggshell added to AA6063 alloy. The densities of AA6063 alloy and calcined eggshell powder were 2.7 and 2 g/cm^3, respectively. Since the density of calcined eggshell (2 g/cm^3) is less than the density of AA6063 alloy (2.7 g/cm^3), the overall density of the AA6063 alloy/eggshell composites is reduced. For the density analysis of AA6063 alloy/eggshell composite four specimen have been prepared with zero percent eggshell, 5 percent eggshell, 10 percent eggshell and 15 percent eggshell. The density of the specimen with five percent eggshell is lower than the density of the specimen with zero percent eggshell. The theoretical density of the composites decreased from 2.7 g/cm^3 to 2.665 g/cm^3, 2.63 g/cm^3 and 2.595 g/cm^3 at 5%, 10% and 15% of carbonized eggshell powder respectively. This shows that the weight of AA6063 alloy/eggshell composite is reduced when calcined eggshell used as reinforcement material as shown from Figure 5.2.

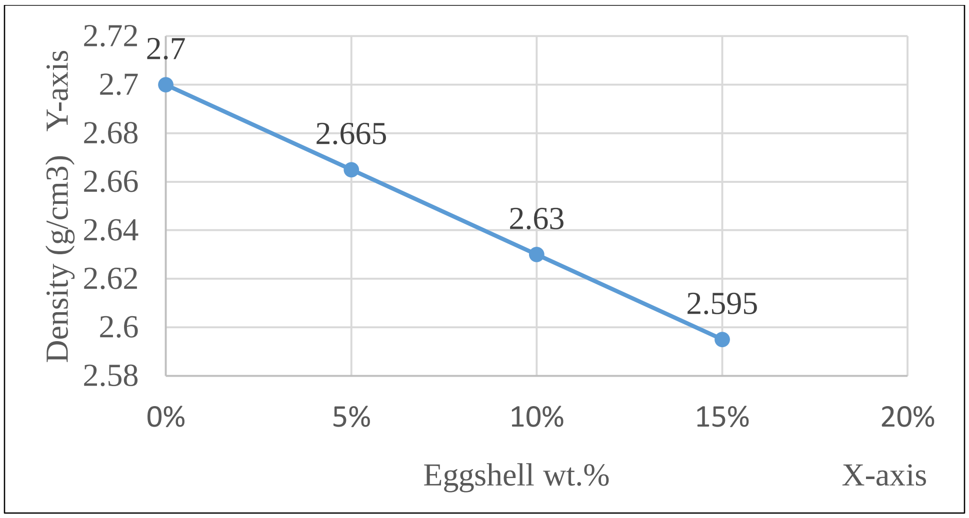

Figure 5.2 Density decrease of eggshell/AA6063 alloy composite

5.8. Tensile Strength and Hardness Test Specimens at Optimum Conditions

The influence of different eggshell weight percentage added to AA6063 alloy can be analyzed by determining the mechanical behavior of these composite specimens subjected to different types of tests. The effect of calcined eggshell powder in AA6063 alloy can be studied by conducting standard mechanical tests on the specimens. In this study, the composite specimens with different eggshell content are analyzed using two important mechanical properties, tensile strength and hardness testing. The specimens are prepared at optimum condition stir casting parameters. The results of specimens with 10 weight percentage of eggshell are obtained from confirmation test.

5.8.1. Tensile Strength of AA6063 Alloy/Eggshell Composite specimens

Table 5.5 presents the results of tensile strength test specimens which were conducted by Universal tensile testing machine.

Table 5.5: Tensile strength specimen results

S.No.	Composition	Tensile strength (MPa)		
		T1	T2	Mean
1	Pure AA6063 alloy	82.14	84.56	83.35
2	AA6063 alloy + 5% eggshell	137.11	135.90	136.505
3	AA6063 alloy +10% eggshell	156.05	152.43	154.24
4	AA6063 alloy + 15% eggshell	129.07	127.44	128.255

It is found that the addition of eggshell powder has a significant effect on the tensile strength. Tensile strength increased from 83.35 MPa at eggshell zero weight percentage to 154.24 MPa at 10 weight percentage for carbonized eggshell powders. The increases in tensile strength with additions of eggshell up to 10 weight percentage are due to the smaller grain formation and uniform distribution of eggshell powders which enhances the tensile strength of AA6063/eggshell composite. It was observed that after addition of eggshell powders more than 10 weight percentage, tensile strength of the composite began to decrease. This occur due to poor distribution, formation of coarse grain, porosity and agglomeration of the eggshell powders in the AA6063 alloy matrix resulting in weak interaction of AA6063 alloy/eggshell composite. This poor eggshell powders distribution, coarse grain size and agglomeration reduces the eggshell powders/AA6063 alloy matrix interaction and as a result decreases the tensile strength of the composite. Figure 5.3 shows that pure aluminum (AA6063) alloy has low tensile strength than AA6063 alloy containing calcined eggshell powders.

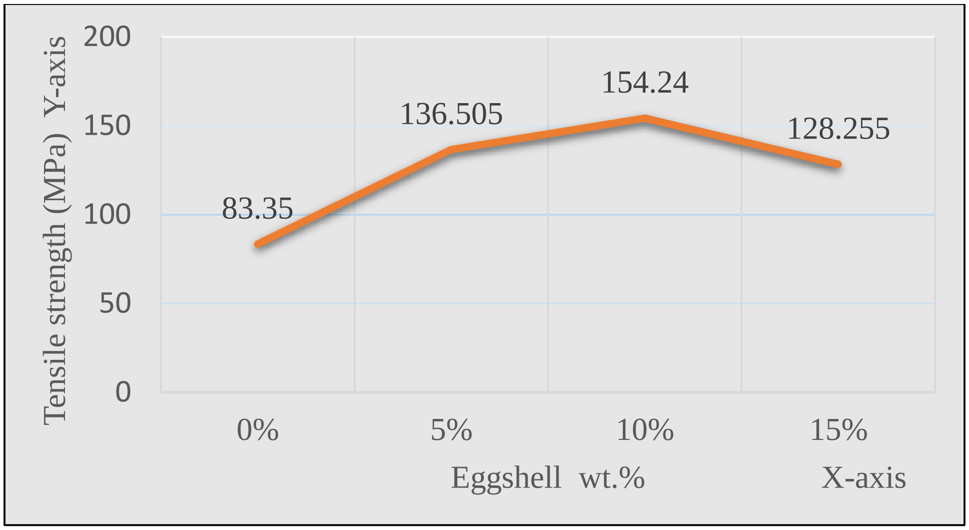

Figure 5.3: Tensile strength vs eggshell weight percentage graphs

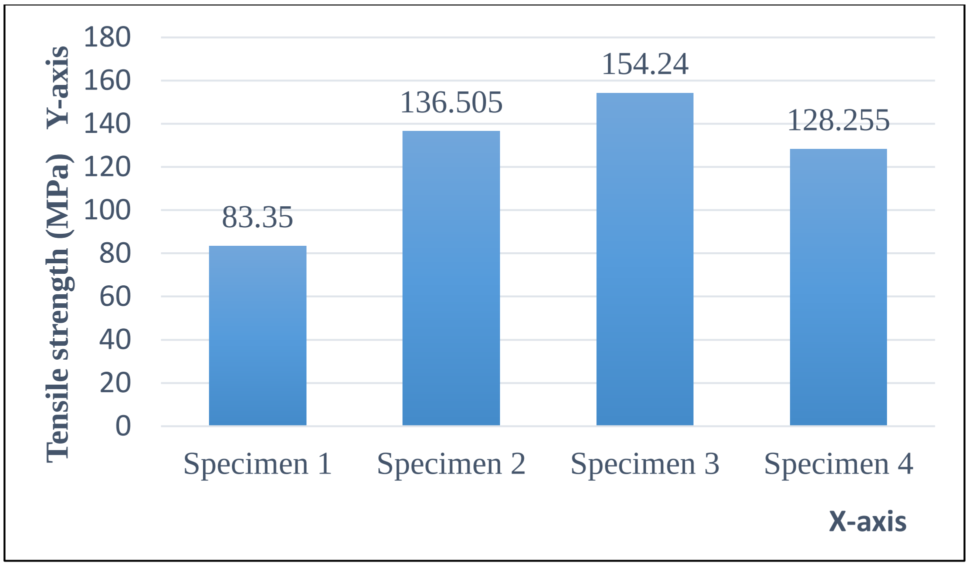

Figure 5.4 comparison of tensile test specimens result

Figure 5.4 shows that the tensile strength of AA6063 alloy/eggshell composite were increased up to 10 weight percentage of eggshell. The tensile strength decrease beyond 10 weight percentage of eggshell powders. Hence from the above graph the tensile strength of the carbonized eggshell/AA6063 alloy composites are more at 10 weight percentage of eggshell powders specimen. This is because of the strong interfacial bonds which improves the tensile strength of carbonized eggshell/AA6063 alloy composite.

5.8.2. Hardness of AA6063 Alloy/Eggshell composite specimens

The property of material to resist against surface deformation or penetration is its hardness. Vickers hardness measurements were carried out in order to investigate the influence of eggshell weight percentage on the matrix metal (AA6063 alloy). AA6063 alloy/eggshell composite containing two different materials (soft AA6063 alloy and hard eggshell particles). The selection of region to be collected in the surface specimen for evaluating the hardness data is very crucial in the case of eggshell-reinforced composites. Some local regions in the composite show remarkable changes. if the eggshell powder accumulated in a particular place the values are higher and if the eggshell powders are absent in some places the values are lower. Therefore, soft matrix, hard reinforcing phase and defects areas should be avoided in order to obtain the average hardness from these tests. The hardness of composite depends on the hardness of the eggshell powders and AA6063 alloy. The hardness measurements were taken in cast specimen to understand the distribution of the eggshell powders in the AA6063 alloy at different processing conditions. Table 4.2 presents the results of hardness test specimens which were conducted by Vickers hardness testing machine.

Table 5.6: Hardness specimen results

S.No.	Composition	Tensile strength (MPa)		
		T1	T2	Mean
1	Pure AA6063 alloy	82.14	84.56	83.35
2	AA6063 alloy + 5% eggshell	137.11	135.90	136.505
3	AA6063 alloy +10% eggshell	156.05	152.43	154.24
4	AA6063 alloy + 15% eggshell	129.07	127.44	128.255

The Vickers hardness (VH) test values for the composite specimens with 0 wt.%-15 wt.% carbonized eggshell powders under the influence of AA6063 alloy is shown in the Figure 5.5.

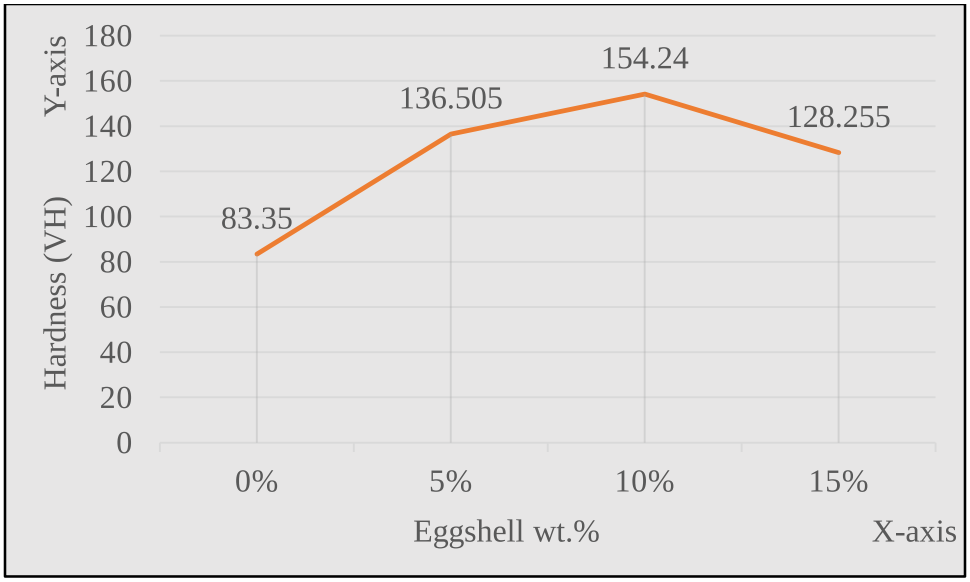

Figure 5.5 Graph of Vickers Hardness Vs eggshell wt.%

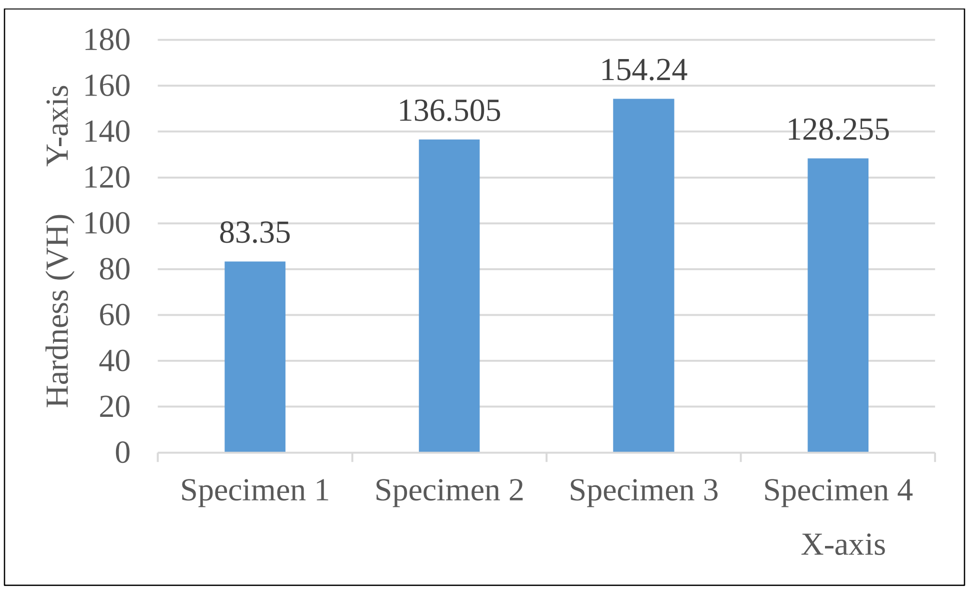

Figure 5.6 comparison of hardness test specimen results

Figure 5.6 shows that pure aluminum (AA6063) alloy having low hardness number and then the hardness number increases as the weight of eggshell particles increases up to 10 wt.% and the hardness number decrease beyond 10 wt.% of eggshell particles. Hence from the above graph the Hardness of the carbonized eggshell/AA6063 alloy is maximum at 10 wt.% of eggshell particles.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1. Conclusion

This paper reports on an experimental analysis and optimization of the stir casting process parameters. The composites containing AA6063 alloy with 0, 5, 10 and 15 wt. % of eggshell powders were successfully synthesized by stir casting technique. AA6063 stir casted metal matrix composite process parameters on tensile strength and hardness values are investigated by Taguchi method. Taguchi's method of design of experiment is a good technique in maximizing tensile strength and hardness of composite materials.

A statistically designed experiment based on Taguchi optimize process was executed by means of L27 orthogonal array to investigate the hardness and tensile strength as response variables. For four parameters and three levels L27 orthogonal array was selected. The combination of parameters and their levels for maximum tensile strength and hardness is $A_2B_1C_2D_2$ (i.e. eggshell 10 wt.%, stirring speed 200 rpm, stirring time 15 minute, and stirring temperature 750 ℃). Analysis of variance determined the contribution of each parameter in the hardness and tensile strength of the prepared composite. The contribution of eggshell wt.%, stirring speed, stirring time, and stirring temperature to the quality characteristics are 50.12%, 10.4%, 27.44%, 8.87% for tensile strength and 68.82%, 7.5%, 15.69%, 3.88% for hardness.

From the confirmation experiment the predicted values and measured values are fairly close to each other, which indicate that the composite can be effectively developed with 95% confidence levels. The confirmation results revealed that maximization of tensile strength and hardness could be arrived significantly for composite casting. Using such method, a remarkable saving in time and cost has been obtained.

The microstructural analysis of AA6063 alloy composite containing 5% and 10% percentages of carboned eggshell revealed near-uniform distributions of eggshell particles and excellent bonding interface with the AA6063 alloy. Clustering, porosities and non-homogenous dispersion of eggshell particles in AA6063 alloy matrix were observed in the microstructures of 15% eggshell composite. The main considerable factor in this process was to ensure presence of excellent wetting between the eggshell particles and molten aluminum (AA6063) alloy, which enhances the mechanical property of composite materials.

From the results and discussion of this work, the following conclusions are drawn:

- ❖ Stir casting is very efficient, simple and cost effective method of manufacturing of aluminum matrix composites and also it is most suitable for mass production.
- ❖ The aluminum (AA6063) alloy reinforced with eggshell powders at different levels and parameters were successfully produced via stir casting method.
- ❖ Tensile strength and hardness of the composite materials increases with an increase in reinforcement of eggshell powders from 0 wt.% to 10 wt.%.
- ❖ The optimum settings of influential parameters in the stir casting process were experimentally obtained as, 10 wt.% carbonized eggshells, 200 rpm stirring speed, 10 minutes stirring time and 750℃ stirring temperature respectively.
- ❖ Based on ANOVA results, the eggshell wt.% is the most significant factor for maximizing tensile strength and hardness, while stirring temperature is the least important.
- ❖ The significance of the influential parameters in the 95% CL was analyzed, so all parameters have a significant effect on the hardness and tensile strength.
- ❖ The presence of greater amount of carbonized eggshell (greater than 10 wt.%) decrease the values of hardness and tensile strength.
- ❖ The overall theoretical density of AA6063 alloy/eggshell composite decreased with additions of carbonized eggshell particles.
- ❖ The uniform distribution of reinforcement in metal matrix was achieved and examined by optical micrographs.
- ❖ Incorporation of CES powders in the aluminum (AA6063) alloy can lead to the production of low cost aluminum composites with improved mechanical properties (hardness and tensile strength).

5.2. Recommendation

Based on the results and conclusion found from the study, the researcher forwards the following important recommendations:

- ❖ In this study carbonized eggshell powders of 5-15 weight percentage have been used. This can further be extended to other eggshell weight percentage to study the effect of weight percentage on tensile strength and hardness of the composite.
- ❖ The study can be extended by the addition of other low cost reinforcement materials with AA6063 alloy and the mechanical properties studies can also be carried out.

- This work investigation was limited to sand casting only. However other available casting techniques could be tried and analyzed so that a final conclusion can be drawn.
- The researcher does further examination on eggshell powder, which shows a great potential to substitute conventional reinforcement. This can turn waste eggshell into high value products.

5.3. Future Work

- The most effective chemical modification will be done on eggshell powder to make it more reactive with different aluminum alloy.
- Effects of different reinforcement materials on the matrix alloy are the areas which are still to be investigated.
- The effect of mold design, riser design in the production of metal matrix composites can be an interesting field of investigations.
- Fabrication of AMMCs by other advance processes like powder metallurgy, diffusion, deposition, etc. may be done to achieve different set of mechanical properties.

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APPENDIX-A: Analysis of Variance for Tensile Strength

Taguchi Orthogonal Array Design (for tensile strength)

L27(3^4)

Factors: 4
Runs: 27

Columns of L27(3^13) Array

1 2 3 4

Response Table for Signal to Noise Ratios
Larger is better

Level	A	B	C	D
1	41.92	42.63	42.16	42.03
2	43.10	42.01	42.86	42.64
3	41.82	42.21	41.82	42.18
Delta	1.28	0.62	1.04	0.61
Rank	1	3	2	4

Response Table for Means

Level	A	B	C	D
1	125.1	136.2	128.8	126.4
2	143.5	126.3	139.7	135.7
3	123.5	129.6	123.5	130.0
Delta	20.1	10.0	16.2	9.3
Rank	1	3	2	4

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
A	2	2242.0	1121.02	141.16	0.000
B	2	465.0	232.49	29.27	0.000
C	2	1226.3	613.16	77.21	0.001
D	2	396.9	198.47	24.99	0.002
Error	18	142.9	7.94		
Total	26	4473.2			

APPENDIX-B: Analysis of Variance for Hardness

Taguchi Orthogonal Array Design (for hardness)

L27(3^4)

Factors: 4
Runs: 27

Columns of L27(3^13) Array

1 2 3 4

Response Table for Signal to Noise Ratios
Larger is better

Level	A	B	C	D
1	39.60	39.81	39.27	39.38
2	40.43	39.11	40.00	39.76
3	38.32	39.44	39.09	39.22
Delta	2.11	0.70	0.91	0.54
Rank	1	3	2	4

Response Table for Means

Level	A	B	C	D
1	95.71	98.57	92.92	93.38
2	105.43	91.08	100.71	97.73
3	82.73	94.23	90.24	92.77
Delta	22.71	7.50	10.47	4.96
Rank	1	3	2	4

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
A	2	2336.0	1167.98	151.09	0.000
B	2	255.0	127.49	16.49	0.000
C	2	532.6	266.28	34.45	0.001
D	2	131.7	65.87	8.52	0.003
Error	18	139.1	7.73		
Total	26	3394.4			