Addis Ababa Institute of Technology

School of Chemical and Bio Engineering

Synthesis, Parametric Optimization and Characterization of Gum Arabic Powder

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A thesis submitted to the School of Chemical and Bio Engineering, Addis Ababa Institute of Technology, in partial fulfillment of the requirements for the Degree of Master of Science in Chemical Engineering (Process Engineering)

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This is to certify that the thesis prepared by Lubaba Moshe, entitled, "Synthesis, Parametric Optimization and Characterization of Gum Arabic Powder" is submitted in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering (Process Engineering) complies with the regulations of the university. The thesis meets the accepted standards with respect to originality and quality.

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DECLARATION

I, the undersigned, declare that this thesis is my original work, has not been presented for a degree in this or any other university, and all sources of materials used for the thesis have been fully acknowledged**.**

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ABSTRACT

Gum Arabic, or acacia gum, is the resin exuded by *Acacia Senegal* trees. It also has several industrial applications. But these, natural gum Arabic exudates are contaminated by sand, dirt, insects, pieces of bark and leaves, bacteria and so forth. This condition makes the gum exudes to be unsuitable for the immediate use. Therefore, finding the purification process of gum Arabic is the first and the most important step. This mechanism improves the quality of the product and makes direct immediate use of acacia gum in the industry. The core objective of this research work was to produce purified, dried, instant gum Arabic powder and develop optimization of the freeze drying operating conditions. Finally characterization of the powder was carried out and yield was determined. To conduct this study, impurities of gum exudates were removed by three major separation processes. This is carried out by dry cleaning, aqueous solution cleaning and freeze drying process respectively. In this study, different concentrations of dissolved gum Arabic solution and drying temperature of 25%w/v to 33.33%w/v and -50°C to -30°C as maximum and minimum values were used respectively. Optimization for these drying parameters was carried out by the central composite design, using the Response Surface Methodology (Design experiment software version 11). During the experiment, it was observed that the acacia gum yield at different runs ranged from 96.35g to 214.1g and the moisture content ranged from 6.07% to 13.9%. An optimum acacia gum yield of 136.12g and minimum moisture content 10.16% were observed at -31.8°C temperature and 33.33% (w/v) feed solution concentration. The two main factors considered for this research study, namely feedstock concentration and drying temperature had a statistically significant effect ($p<0.05$) on both responses named the powder yield and moisture content. After the synthesis, the product (acacia gum powder) was characterized using FTIR spectroscopy, NMR Spectroscopy, TGA Analysis and DSC Thermogravimetric Analysis.

Keywords: Gum Arabic powder, Freeze drying, Moisture content, Yield, Separation process, Response surface methodology, Central Composite Design, Optimization.

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ACRONYMS AND ABBREVIATIONS

GA	Gum Arabic
g	Gram
NWFP	Non-Wood Forest Product
JECFA	Joint Expert Committee on Food Additive
ASAL	Arid and Semi Arid Lands
GRAS	Generally Recognized As Safe
FAO	Food and Agricultural Organization
TGA	Thermo Gravimetric Analysis
DSC	Differential Scanning Calorimetry
FT-IR	Fourier Transform Infrared Spectroscopy
NMR	Nuclear Magnetic Resonance
ANOVA	Analysis of Variance
BBD	Box -Benkhen Design
CCD	Central Composite Design
RSM	Response Surface Methodology
DOE	Design of Experiment
RP	Reverse Phase

CHAPTER ONE

1. INTRODUCTION

1.1. Background

Acacia Gum or Gum Arabic is a natural agricultural resource from the Gum Belt region of Africa, i.e. countries geographically ranging from East to West - from Sudan, Somalia, Eritrea and Ethiopia to Chad, Central African Republic, Mali, Niger and further west up to Nigeria, Senegal and even Mauritania. Economically speaking, Acacia Gum mainly comes from Sudan, Chad and Nigeria(Sara et al., 2016).Within commercial circles, it is known also as Gum Acacia, Turkey Gum, and Indian Gum and by many other descriptive and colorful local names (Tieguhong and Ndoye, 2004). Originally called Gum Arabic because it had been shipped from Arabian ports to Europe and it is also known as Acacia Gum due to its botanical origins (Sanchez et al., 2018).

During ancient Egypt Acacia Gum has been trade since the most remote records of historical antiquity. A representation of the Acacia tree, together with heaps of gum, was picturing during the reign of Ramses III in Egypt. Not only the Egyptians used it as an adhesive for papyrus sheets, but also as a fixative for the ink on the papyrus or to coat, the mummy bandages. Acacia exported from the Gulf of Aden, seventeen hundred years before Christ. Mention of the gum is of frequent occurrence in Egyptian inscriptions, where it was refer to as the Gum of Canaan. Theophrastus, in the third and fourth centuries before Christ, described it, as also did dioscorides and Pliny, under the name "Egyptian Gum". During the middle Ages Acacia Gum had been employ in the arts from all time and in domestic medicine and commerce, as well as by the Arabian physicians and those of the renowned school of Salerno. Nowadays Acacia Gums are widely used in the food and pharmaceutical industry (ElKhawad, 2008).

In Sudan, the most important NWFP is Gum Arabic, which exudates from acacia Senegal known as hashab or hard gum and acacia seyal known as talh or friable gum (Abdel, 2007).Both species spread naturally in the central belt of the low rainfall savannah where they existed in pure or mixed stands, in the clay plains in the East and sandy soils in the West. Besides its significant economic role for the country, it plays an important part in rural life, providing a steady income to rural families especially in dry years when crops fail (Abdel, 2007).

Sudan has dominated world production and trade of Gum Arabic and during the period (1925-1985) exported from Sudan accounted for around 80% of the world exports. This domination however, has become less marked in recent years. The Gum Arabic Company (28.2 % owned by the government) is the monopoly holder for the export of crude Gum Arabic from the Sudan.

Gum Arabic is one of the main agricultural export commodities produced in traditional rain-fed agriculture in Sudan (Mohammed and Roehle, 2011) and it produced mostly by small farmers in traditional rain field farming areas. These farmers represent up to 20 percent of Sudan's population and are among the poorest. The impact of the current Gum Arabic marketing policy has not been beneficial to this group, but also affected in production and consequently exports, declining for the past forty years at an average rate of 2.2 percent annually (ElKhawad, 2008).

The development of the processing industry over the last three years has resulted in increased domestic competition for raw gum, and in turn better prices paid to farmers as well as more value added captured in Sudan. This positive development came at a propitious time as increased consumption of soft drinks and confectionary products, as well as rapid development of health and dietetic products is boosting the world demand for Gum Arabic (ElKhawad, 2008).

But after gum Arabic is exuded from the Acacia tree within a short period of time, the outer layer of the gum tear becomes contaminated with foreign substances. Such as fine Sand, dirt, insects, pieces of bark and leaves, bacteria and the like. The contaminated outer layer of the gum tear hardens as a result of air oxidation. Therefore, much of the gum Arabic which has been available for transshipment to World markets has been discolored coated with undesirable Surface impurities and otherwise unfit for immediate use. Because of this serious problem, there should be industrially processed mechanism to purify the contaminated gum exudates. This industrially processed condition leads immediate use of gum Arabic for different application especially in foods and pharmaceutical industries (Agazarian et al., 1970).

Freeze-drying is a process in which water is sublimated by the direct transition of water from ice to vapor, thus omitting the liquid state, and then desorbing water from the dry layer. It is widely used for the stabilization of high quality food, biological materials and pharmaceuticals such as proteins, vaccines, bacteria and mammal cells. In the process, the quality of the dried product such as biological, nutritional and organoleptic properties are retained. This is due to the fact that freezing water in the material prior to lyophilization inhibits chemical, biochemical and microbiological processes. Therefore, the taste, smell, and content of various nutrients do not change. Raw food materials contain a lot of water, ranging from 80% to 95%. The removal of water by sublimation results in the creation of highly porous structure of the freeze dried products and the rehydration of lyophilisates occurs immediately (Flink, 1977a; Flink, 1977b, Nowak and Jakubczyk, 2020).

The effect of freeze-drying should be considered from the economic aspect and the quality of the freeze-dried material. The cost of a product mainly depends on the freeze-drying time. Therefore, process parameters and other conditions of its course are often set so that its time is as short as possible. Setting parameters to speed up the process can lead to the deterioration of the product's properties. For example, increasing shelf temperature can lead to the defrosting of the product and the collapse of the structure or to the thermal degradation of heat sensitive food ingredients (Nowak et al., 2016).

The conditions of the freeze-drying process should be selected in a way that does not melt the water. Liquid water is the reaction medium and changes the rheological properties of the product. The presence of liquid water during the freeze drying of food products may result in many changes in the composition, morphology and physical properties of foods to shrinkage. It may also reduce the period of ensuring high quality during storage. The color and structural texture properties are crucial in the quality evaluation of food by consumers. Therefore, the dependence of these food properties on the parameters of freeze-drying is extremely important (Franks and Auffret, 2008; Nowak and Jakubczyk, 2020).

The effect of freeze-drying conditions on the nutritional properties, antioxidant activities and glass transition characteristics of different materials can be found in the literature. It is widely believed that freeze-drying is the best method of drying. However, improperly selected process parameters may cause unfavorable changes in the material such as shrinkage, color change and collapsed structure. Therefore, this study was conducted to optimize the freeze drying temperatures and concentration of feed solution on the gum Arabic powder extraction by using response surface methodology, which is the focus of this study.

1.2. Statement of the Problem

Even though acacia gum has numerous industrial applications, naturally gum Arabic exudates are contaminated by sand, dirt, insects, pieces of bark and leaves, bacteria and the like. To use this natural resource to different types of industrial application, findinging gum Arabic purification mechanism is the first and the most important step. This mechanism improves the quality of the product (Agazarian et al., 1970).In this study, the main target focus on inventing the mechanism of producing pure dried gum Arabic powder and developing optimization of the freeze drying operating conditions.

In Ethiopia, there is availability of raw material and manually cleaning acacia gums which are simply cleaned by hand pick selection of the impurities to remove small bark fibers. And they are exported to developed countries (Tadese et al., 2018). But such types of acacia gum are still contaminated. They need secondary purified processed mechanism to use them in food and pharmaceutical industry. In the world market, pure dried acacia gum powder and the manual cleaned gum has huge difference in selling price of cost. Based on this point of view it is suggested that in future Ethiopia would export the dried powder than exporting the raw acacia gum as it is. This research shows the possibility of producing gum Arabic powder in Ethiopia. Based on this study it is possible to get financial profit which supports the economic growth of Ethiopia.

1.3. Objective

1.3.1. General Objective

The general objective of this work was to produce gum Arabic powder, to optimize freeze drying operating conditions and to characterize the powder.

1.3.2. Specific Objectives

The specific objectives of this study was to:

- To prepare gum Arabic powder from locally available *Acacia senegal.*
- To investigate the process variables (concentration of the solution and freeze drying temperature of the dryer) during the process.
- To optimize the operating conditions to get a maximum yield on the percent of powder recovery and to get minimum amount of moisture content in the powder.
- To characterize the properties of the prepared powder (NMR spectroscopy, FTIR, TGA analysis and DSC thermogram).
- To study numerous mechanism involved to purify the naturally contaminated raw material of gum exudates.

1.4. Significance of the Study

The importance of this study will be directly related to the numerous uses and application of acacia gum. Especially gum arabic powder has significant application in food industry, paint industry, paper print enterprises and pharmaceutical industry as stabilizer, emulsifier, thickening agent, binder, lithographic process and the like (Agazarian et al, 1970; ElKhawad, 2008; Sanchez et al., 2018). Even though there are different types of acacia gum in the world market, the dried powder is the desired and the most expensive grade type. Based on this point, this study highly signifies the possibility to produce gum arabic powder in Ethiopia. Currently the Ethiopian agricultural research institute exports the raw gum (Tadese et al., 2018) and our country again imports the processed one within expensive cost. This study offers acacia gum processing technology suggestions to Ethiopian agricultural research institute about exporting the acacia gum powder. This suggestion finally results attaining sustainable gum Arabic powder production, creating job opportunity for the community around there, saving foreign currency, minimizing the import of proceed acacia gum, support the country's economy and so forth.

1.5. Scope of the Study

This study was conducted in a laboratory scale mainly by using mini freeze dryer as major fundamental equipment. Since freeze dried materials are directly impacted by the effect of freeze drying conditions of the selected physical properties of materials such as drying temperature, freezing rate and working pressure and so forth (Nowak and Jakubczyk, 2020). This thesis research was restricted to the investigation of only two major factors namely drying temperature and concentration of the feed solution.

CHAPTER TWO

2. LITERATURE REVIEW

2.1. Production of gum Arabic

Gum Arabic is the dried, gummy exudation obtained from various species of acacia trees of the leguminous family (Abdel, 2007). The JECFA defined Gum Arabic as dried exudates from the trunk and branches of *Acacia senegal* or *Acacia seyal* of the family Leguminous (Rahim et al., 2010). Acacia trees belong to the botanical family of Leguminous, predominantly species of the groups Fabales and Gummiferae. There are about 1100 several species of acacia gum distributed over tropical and sub-tropical areas of Africa, India, Australia and America. However, only *Acacia senegal* (high quality gum) and *Acacia seyal* (low quality) yield gum of economic significance (Mahmoud et al., 2014).

Acacia senegal is a small to medium sized thorny tree, with a stem, which is irregular in form and often highly branched. In leaf, like many other Acacias, it has a dense, spreading crown. The bark is not papery or peeling. The tree is deciduous, droops its leaves in November in the Sudan (Ndoye and Tieguhong, 2004). Acacia seyal has a single straight stem with a characteristic, pronounced color, usually orange-red, to the powdery bark, and straight thorns rather than the curved prickles of acacia Senegal.

Gum production evolving from tapping in the wild to a scientific agro foresting operation providing higher and more reliable yields as well as better quality of production. Tapping, Harvesting and cleaning are major steps to collect the raw material of acacia gum (ElKhawad, 2008). Tapping involves removing sections of the bark and wound (sticky gum substance dries on the branch of tree to form nodules called kibble) with an axe. Harvesting involves gum exudates picked by hand will be placed in an open plastic basket carried by the collector. Cleaning involves removing impurities, sands,pieces of bark and then put in the gum under sun light. The amount produced gum varies, however, from 20g to 2000g depending on the tree; on average, a tree yields 250g.

Table 2-1: Factors affecting the production of gum Arabic

Factors	Beneficial Effect	Adverse Effect
Rainfall	Good rain is a signal of good gum production(400 to 900 mm per annum/season)	Continuous rainfall or showers very late is bad for gum production
Drought	Drought immediately after the end of the rainy season improves gum production	Gum gardens or natural stands wiped out by drought
Temperature	High temperature is conductive to good production if the trees are tapped at the right time	Low temperature at tapping time slows gum exudation, sometimes tree tapping has to be repeated
Size and range of the trees	Mature trees (15-20 years old)	Young trees (<5) and very old ones (>20) years old are low producers
Health of the trees	Trees with good growth during rainy season have high production	Trees with poor growth during the growing season have low production
Injector insect (Garraha)	Farmers believe that garraha increases production	--
Animals	Cattle and sheep are grazers, keeping the vegetation down reducing or even eliminating fire hazards	Animals graze on the leafs of the acacia trees (particularly goats and camels)
Fire	--	Fire damages live trees and destroys all seeds fallen on the ground
Expansion of agriculture	--	Gum gardens expand their cash crop production which reduces gum production
Locusts	--	Tree locust and desert locust attacks result in partial/complete gum production failure
Shortage of water supply	--	Drinking water shortages and long distances for water supply for tappers, indirectly affect production quantity

Rural poverty	--	Increased internal migration to the cities subsequently decrease gum production
Marketing and transport	Adequate marketing cycle and transportation will lead to production sustainability	Lack of adequate marketing cycle and transportation on gum belt results in unstable sustainability of supply

2.2. The Gum Arabic Belt

The gum belt referred to a broad band, situated at a latitude of between 12° and 16° North, stretching across sub-Saharan Africa, from Mauritania in the West, through Senegal and Mali, Burkina Faso, Niger , Northern Nigeria to Sudan, Eritrea, Ethiopia, Kenya, Somalia and Northern Uganda in the East as shows in (figure 2-1) (Abdellah, 2019).Production of Gum Arabic is concentrated in the "gum belt", an area of central Sudan roughly between latitudes 10° and 14° north. Two areas outside these borders are in the north east (Faw-Gedared-Kassala) and in the south east along the Blue Nile/Upper Nile border (ElKhawad, 2008).

Sudan gum Arabic belt estimated to cover 520.000 square kilometers, roughly one fifth of Sudan's total area. It spanned over 12 states:-Western Darfur, N. Darfur, S. Darfur, N. Kordofan, W. Kordofan, S. Kordofan, White Nile, Upper Nile, Jonglie, Sennar, Blue Nile and Gedaref. The sandy plains are in the first seven states and the clay plains are in the latter five states as shows in (figure 2-2) (Abdel Nour, 2007).

Acacia Senegal has a wide distribution and remarkable adaptability. It is essentially a semi-arid zone species, but it is both drought, frost resistant, and can grow with a rainfall of between 100 and 800 mm per year. It grows across Africa, from Senegal to Ethiopia, through Mali, Nigeria, Chad and Sudan, to mention only the major producing areas. It is also found in the Middle East, Yemen, India and Pakistan (Islam et al., 1997).

In the Sudan, particularly in the Kordofan and Darfur provinces, the species is uniform and found in pure stands giving the Sudan an important advantage of being the most important producer of this type of Gum Arabic. In other producing countries, acacia Senegal often found mixed with other species. Another feature of the Sudan system of production is that this species occurs both as a wild and as a cultivated species; man in village plantations often replants it (ElKhawad, 2008).

Acacia Seyal grown in the Sudano-Sahelian belt where the rainfall is slightly higher than in the regions populated with the acacia Senegal. It grown and regenerated naturally; it does not required tapping and gum exudes naturally. Its biomass calculated to be 50 times greater than that of the acacia Senegal, which gave it an important advantage in terms of production potential. It affected at a later stage than the acacia Senegal by the desertification process. It can grow on clay soils and resists well to climatic changes from temporary wet to prolonged dry periods with a consequent cracking of the surface of the soil (ElKhawad, 2008). Whilst the acacia Senegal and the acacia seyal are present together in the same geographical zone, they have their own biotope (for example, nearer to water for the acacia seyal, drier areas for the acacia Senegal) (Abdellah, 2019).

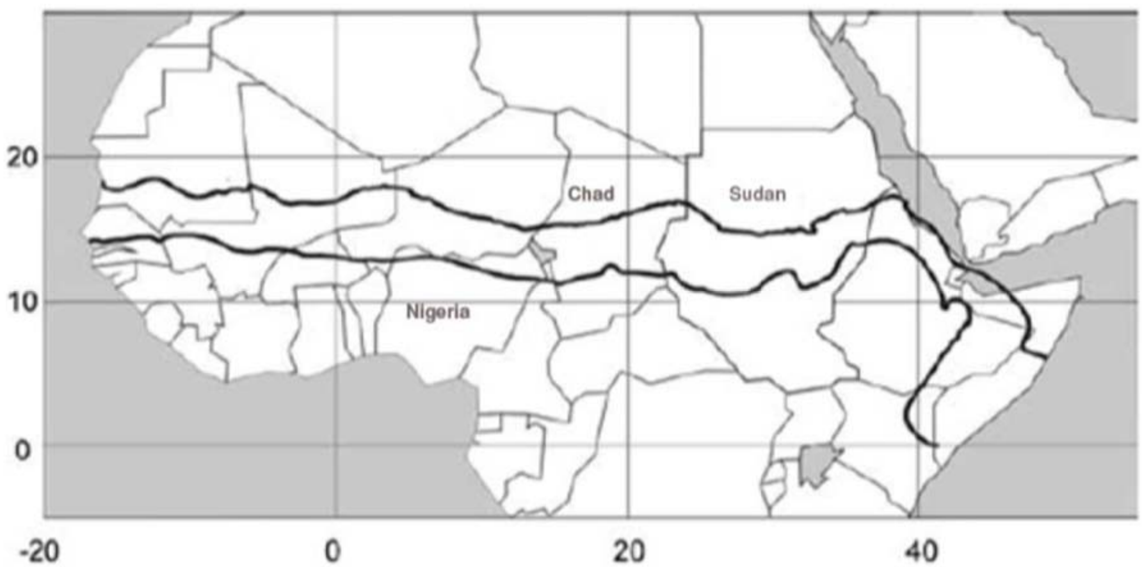


Figure 2-1: The gum Arabic belt in Africa

There are close to 1200 acacia species capable of producing gum (Ross, 1979).These are primarily located in tropical climates, with about 130 of them located specifically on the African continent. Africa, therefore, quickly became the major site of the production of gum; this is the reason why it is also referred to as “Senegal Gum”. Gum is essentially the secretion of several acacia (leguminous) trees. Acacia Gum species, of which there are up to seventeen, produce acacia gum of varying quality and quantity (Islam et al., 1997).

Kenya is richly endowed with Acacia trees. It is at the crossroads of two gum Arabic belts; one running just south of the Sahel, the other along the east coast of Africa. Nearly 80% of the country's area contains trees that produce gum Arabic (Maundu et al., 1999), most of which are of the species Acacia-Senegalvarkerensis(Booth and Wickens, 1988). However, Kenya exports only small amounts of gum Arabic. Kenya is currently a new supplier of gum Arabic in the world market, but the country does not meet the competitiveness and adequate supply of the commodity to the market. Because of problems relating to qualities, Kenya's gum is not able to attract premium prices compared with the Sudan gum in the world market.

Ethiopia also has widely differing agro-ecological zones, commonly classified into highlands – areas above 1500metres above sea level – and lowlands, those below. Arid and semi-arid lands (ASALs), a class of dry lands, are important in both the highlands and the lowlands of Ethiopia. These areas are povertystricken and largely food insecure, despite being endowed with resources that could provide alternative and sustainable livelihoods if they were properly exploited and developed. Vegetation in Ethiopia's ASALs includes diverse plant species that produce commercially important oleo-gum resins such as Gum Arabic, Frankincense, Myrrh and Opoponax.

Figure 2-2: The Sudan Gum Arabic Belt

2.3. Industrial applications of Acacia gum

Since gum Arabic is nontoxic, odorless, has a bland taste and it does not affect the odor, color or taste of the system in it, acacia gum is in the GRAS (General Recognized As Safe) list under the Federal Food, Drug and Cosmetic Act. Because of this it can be used in the following industries. In food industry acacia gum used in Confectioneries as prevent crystallization of sugar, Dairy Products as stabilizer in frozen products, Bakery products as its viscosity and adhesive properties, Beverages as foam stabilizer in beer, and as a clouding agent to give opacity, Flavor emulsifieras emulsifier and protective collide and also it is used in Diabetic and dietetic product because of its low level of metabolism (McNamee et al., 1998). In pharmaceutical industry it is used like Emulsions: as a stabilizer, Tablets: as a binder, Tablet coatings: as mucilage, Cough drops and syrups: as an emollient and demulcent (Joseleau and Ullmann, 1990). In cosmetic industry it isapplied in Lotions and protective creams: Used to give smooth feel, Facial masks: as an adhesive, Face powders: as an adhesive.In textile industry acacia gum used as sizing and finishing agents and in printing formulations for imparting designs and decorations to fabrics, also used to thicken the dye baths that are used in the printing and dyeing of fibers, fabrics and carpets(Anderson, 1978). In paint industry it is used: as a protective collide, as a flocculants and emulsifier in vinyl resin emulsions. For ink: Used as a protective collide and suspending agent. In lithography: Used as a sensitizers for lithographic plates. On Other Industries gum Arabic used in glue for paper, glass and metal products (Idris et al., 1998).

2.4. Arabic gum powder production methods

2.4.1. Mechanically removing the impurity

This mechanism is also known as dry cleaning step. This is done mainly to remove impurities containing outer layer from partially hardened gum Arabic. This step involves Hand-picked selected (primary cleaning) to obtain expensive grade which is cleanest lightest color in the form of large nodules. Tray cleaning to remove impurities that remains after hand-picked selection. Vibrated screening is done to remove sand sifting gum. Magnetic detector is used to remove metals remain in the material. Crushing is done to reduce the size of gum and grind the remaining sand and bark in order to remove easily by air cyclone. Air cyclone is used to remove the grinded sand and bark. Course crushing is done to make that the grinded gum may not have uniform size so, this may give same size. Finally Sieving is done to remove small barks remained after the above process (Agazarian, 1970; Meda and Ratti, 2005; ElKhawad, 2008).

2.4.2. Solubilizing the purified gum in a fluid

This mechanism is known as aqueous solution cleaning step. This is done mainly to form a solution free of fluid-insoluble impurities. This step involves dissolving in water to make easy purification process and fine impurities are removed by the next process. Vacuum filtration is done to remove impurities of bark and sands by using special filter cloth. Centrifugation is done next to settled and collected dust and different impurities in one side of the device due to density difference. Pasteurization is done lastly to remove growth of micro-organisms (Agazarian, 1970; ElKhawad, 2008).

2.4.3. Freeze drying the solubilized gum Arabic solution

This is done lastly where a sterilized, highly purified and dried gum Arabic is formed by drying the solution in the freeze dryer. In freeze drying process, all ice water and some bound water must be removed. The water in the products can be free water or water bound to the matrix by various forces. Therefore, lyophilization is a highly complex and multi step process that consists of three steps. Which are freezing of the product under atmospheric pressure, primary drying involving ice sublimation at reduced pressure and secondary drying(desorption drying) that is drying the product to the required final humidity (Ceballos et al., 2012; Nowak and Jakubczyk, 2020).

2.5. Effect of Freeze Drying Conditions for Acacia Powder Synthesis

Freeze dried products are directly influenced by a set of freeze dryer parameters, such as the drying temperature, freezing rate and pressure chamber (Nowak and Jakubczyk, 2020). Additionally, a variety of feedstock specificities and process parameters also play a crucial role in the final particle characteristics, conferring different morphologies, sizes or residual moisture amounts. It is thus fundamental to realize how these variables influence the drying mechanism in order to achieve an optimized operation (Anandharamakrishnan, 2017).

Drying temperature: The process parameters of freeze drying may significantly affect many quality attributes of foods and other materials subjected to this kind of dehydration. The temperature of the heating plate is one of the parameters that plays an important role in creation of the material structure during the freeze drying. Undesired changes in the material structure can be a result of inappropriate temperature applied in the process (Duan et al., 2016). Therefor drying temperature must be selected based on the feedstock property (Nowak and Jakubczyk, 2020).

Working pressure: The application of adequate working parameters of lyophilization, including the working pressure, may significantly affect the course of the process but also the quality of the obtained products (Martinez et al., 2019). Pressure has a strong impact on the sublimation process, especially the temperature of ice sublimation. The sublimation removal of ice creates pores and gaps with different characteristics. Therefore, the desired porosity, density, and final water content can be obtained by manipulating the freeze-drying parameters (Li et al., 2014; Oikonomopoulou et al., 2011).

Freezing Rate: The effect of the freezing rate on the properties of freeze-dried products could be related to the initial structure and composition of materials. The higher freezing temperatures resulted in less rapid nucleation and a longer time of ice crystal growth, which led to the occurrence of large pores (Liapis and Bruttini, 1994; Nowak and Jakubczyk, 2020).

2.6. Determination of Gum Arabic

Qualification refers to the degree to which an object or entity (e.g., process, product, or service) satisfies a specified set of attributes or requirements. It is the degree to which a set of inherent characteristics fulfills requirements (Liu et al., 2008). Therefore, for each product there are certain qualifying indices or parameters that should be assured before the use or application (Rossiter, 2002). The quality of gum Arabic as received by the importer is very dependent on the source. Gum Arabic (hashab) from Sudan is the highest quality and sets the standard by which others types of “gum Arabic" are judged. Not only does Sudanese gum come from a species (*A. senegal*) which intrinsically produces a high quality exudate with superior technical performance, but the collection, cleaning, sorting and handling of it up to the point of export is well organized and highly efficient. Analytical data for the gum obtained from Acacia Senegal shows 12.5–16.0 %, 32.7–27.0 %, 0.22–0.39 %, 1.5–2.6 %, 39–42 %, 24–27 %, 12–16 %, 15–16 %, 1118–1,238 Da for moisture content, specific rotation, nitrogen, protein, galactose, arabinose, rhamnose, glucuronic acid, equivalent mass respectively. In the past, gum Arabic was sold only as one grade (cleaned). Nevertheless, from1995 it has been cleaned and sorted into three grades: Super, Standard clean and Siftings (ElKhawad,2008; Rudy et al., 2015; Sanchez et al., 2017).

For the case of freeze dried gum Arabic the quantification and identification of the product mainly depend on the effect of freeze dryer conditions which are mentioned on the above topics. Which are effect of freeze drying conditions of gum arabic powder production (Nowak and Jakubczyk, 2020). There are large numbers of spectrometric and thermal analysis methods that have been used for the quantification and identification of freeze dried gum Arabic. With the advent and advancement of analytical science, there has been an array of modern tools, such as FT-IR, NMR, TGA and DSC amongothers (Jenke, 1996; Sara et al., 2016). The Percent of powder recovery is a measure of the solution's efficiency to produce freeze dried gum Arabic from the original aqueous solution. It was defined as the amount of powder recovered in mass compared with the initial amount of the sample. It is presented in percentage and it should be determined for each experimental run. The obtained powder is finally weighed to determine the percent of powder recovery (Alsadon et al., 2023).

2.7. Optimization

Optimization describes the process of finding the optimal solution for a certain problem that is characterized by a specific model and a corresponding objective. It is the selection of process controls and operating parameters that add the largest value that is available with existing resources. It involves an act, process, or methodology of making something (such as a design, system, or decision) as fully perfect, functional, or effective as possible. It refers to improving the performance of a system, a process, or a product to obtain the maximum benefit from it. The term optimization has been commonly used as a means of discovering conditions at which to apply a procedure that produces the best possible response (Araujo and Brereton, 1996). When optimization has been carried out by monitoring the influence of one factor at a time on an experimental response, only one parameter is changed, others are kept at a constant level. This optimization technique is called one variable at a time. Its major disadvantage is that it does not include the interactive effects among the variables studied. Consequently, this technique does not depict the complete effects of the parameter on the response (Lundstedt et al., 1998). Another disadvantage of the one factor optimization is the increase in the number of experiments necessary to conduct the research, which leads to an increase of time and expenses as well as an increase in the consumption of reagents and materials.

2.7.1. Response Surface Methodology Approach

To overcome the problem of one variable at a time optimization technique described above, the optimization of analytical procedures has been carried out by using multivariate statistical techniques. Among the most relevant multivariate techniques used in analytical optimization is response surface methodology (RSM). RSM is a collection of mathematical and statistical techniques based on the fit of a polynomial equation to the experimental data, which must describe the behavior of a data set with the objective of making statistical previsions. It can be well applied when a response or a set of responses of interest are influenced by several variables. The objective is to simultaneously optimize the levels of these variables to attain the best system performance.T his is because statistical tools such as RSM recognize the interaction amongst the parameters and reduce their estimation error (Myers and Montgomery, 2002). These attributes favor the RSM as a better method to use when investigating the effect of operating variables during the production. In the RSM approach, statistical design of experiments (DOE) obtained via a standard design method are conducted systematically. Examples of the standard DOE are full factorial, Box-Behnken Design (BBD), and central composite design (CCD). The BBD uses a spherical design with good certainty within the design space. It requires fewer experiments compared to other DOE and is rotatable or almost rotatable despite the number of variables under investigation (Nath et al., 2008). Besides, BBD is purposely built to fit a quadratic model. It does not have runs at the extreme combinations of all the factors, but compensates by having better prediction precision in the center of the factor space.

CHAPTER THREE

3. MATERIALS AND METHODS

3.1. Raw Materials Collection, Storage and Pretreatment

The raw materials used for this experiment were: Raw gum Arabic provided by Ethiopian Agricultural Products Research Co-operation, Adama, Ethiopia. The raw gum arabic was exposed to sunlight for about 8h and the impurities were initially hand picked. Finally, it is stored in polyethylene bag at room temperature in a dry place to avoid capillary tissue rise due to humidity until it was used for the preparation of the finally desired product.

3.2. Equipment

Major equipments that were used in the whole experimental works were: Plastic bags, Crushers, Sieves, Digital pH meter, Bucket, Vacuum filter, Centrifuge (Labtech, model AVI-558), laboratory autoclave, Refrigerator (WestPoint, model WRES-358.X), Digital balance, Filter cloth, Metal detector, MB45 Moisture Analyzer, NMR Spectrophotometer, Volumetric flasks, Grinder, Stove, Tray, Spatula, Perkin Elmer FTIR Spectrometer(Perkin Elmer, 65, USA), Stirrer, NS 24/29 round bottom flasks, Towel, mini laboratory freeze dryer(FD-12-MTP), DSC- TGA Thermo gram analyzer (Instrument: SDT Q600 V20.9 Build 20).

3.3. General Frame Work of the Experiments.

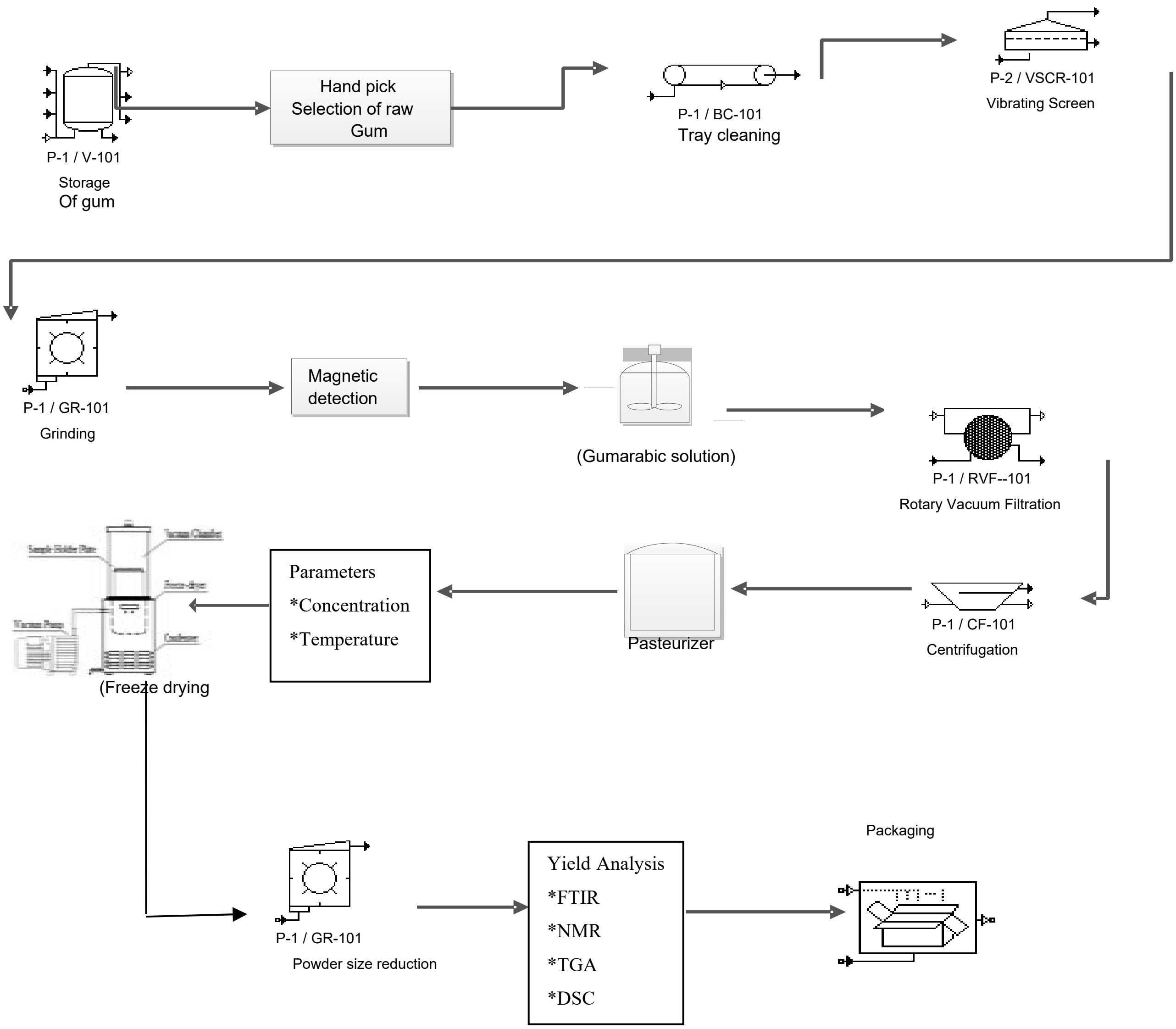


Figure 3-1: Overall framework of the experiment

3.3.1. Preparation of gum Arabic aqueous solution

Aqueous gum Arabic was prepared by following the laboratory procedures. The impurities of contaminated gum Arabic was picked by hand to obtain the most expensive grade which is cleanest, lightest in color and in the form of large nodules. Vibrating screens were used to remove the sand sifting gum. The magnetic detector was used to remove metals remaining in the material. Then the cleaned gum Arabic was crushed to reduce the size of gum and the remaining sand and bark were removed by air cyclone which was used to remove the ground sand and bark. Then coarse crushing was carried out to obtain all gum Acacia with uniform size. Sieving was carried to remove the dusts remaining after the above process. Finally dissolving the gum Arabic in water was carried out (Agazarian, 1970). This step was carried out to make the purification process easy and the fine impurities are removed in the next process. During this procedure, different types of solution were prepared by having concentration differences from 33.3% w/v to 25% w/v as high and low values respectively. Then vacuum filtration was done by using filter cloth to remove impurities. Next centrifugation was carried out. Lastly, the solution was pasteurized to remove micro-organisms growth at 65 °C for 30 min.

Hand-picked selection

↓

Vibrated screening

↓

Air cyclone

↓

Course crushing

↓

Sieving

↓

Dissolving in water

Figure 3-2: Flow diagram of preparing gum Arabic Aqueous solution.

3.3.2. Freeze drying Process

Freeze dryers work by freezing the material, then reducing the pressure and adding heat to allow the frozen water in the material to change directly to a vapor. In this study, freeze-drying is preferred because it removes the water content without greatly altering the physical structure of the resin. The prepared solution was initially frozen in the lab freezers before they were subjected to freeze-drying. The feed solution was then placed in the round bottom flasks of the freeze dryer containers to allow as much of the sample to be exposed to the vacuum as possible. The freezer unit was switched on and left for about 20 min to allow the stabilization of the temperature to stabilize around -45 °C. During freeze drying, the feed solution was subjected to the freeze-drying process in the round bottom flasks by keeping all other parameters constant in all trials except drying temperature and feed concentration.

Preparation of feed solution concentrations

↓

Homogenizing

↓

Freeze drying

Drying temperatures

↓

Freeze dried acacia gum

↓

Characterization

Figure 3-3: Flow diagram for freeze drying of gum Arabic Aqueous solution.

The measured amount of solution of different concentration was then poured into the round bottom flask for each trial and freeze drying was carried out in the subsequent steps. Then the vacuum pump is switched on by ensuring that the drain valve is closed and the flask unit is sealed. After the temperature has stabilized and the solution is heated, the pump was switched off by leaving the freezer on. The vacuum is then released by opening the drain valve. After the vacuum has been released, the dried samples were taken from the freezer and transferred to the flask as quickly as possible. After drying, both the vacuum pump and the freezer unit is switched off. Finally, the dried acacia gum were recovered and then the amount were weighted by digital balance for all trials and placed in sealed plastic bags for further analysis.

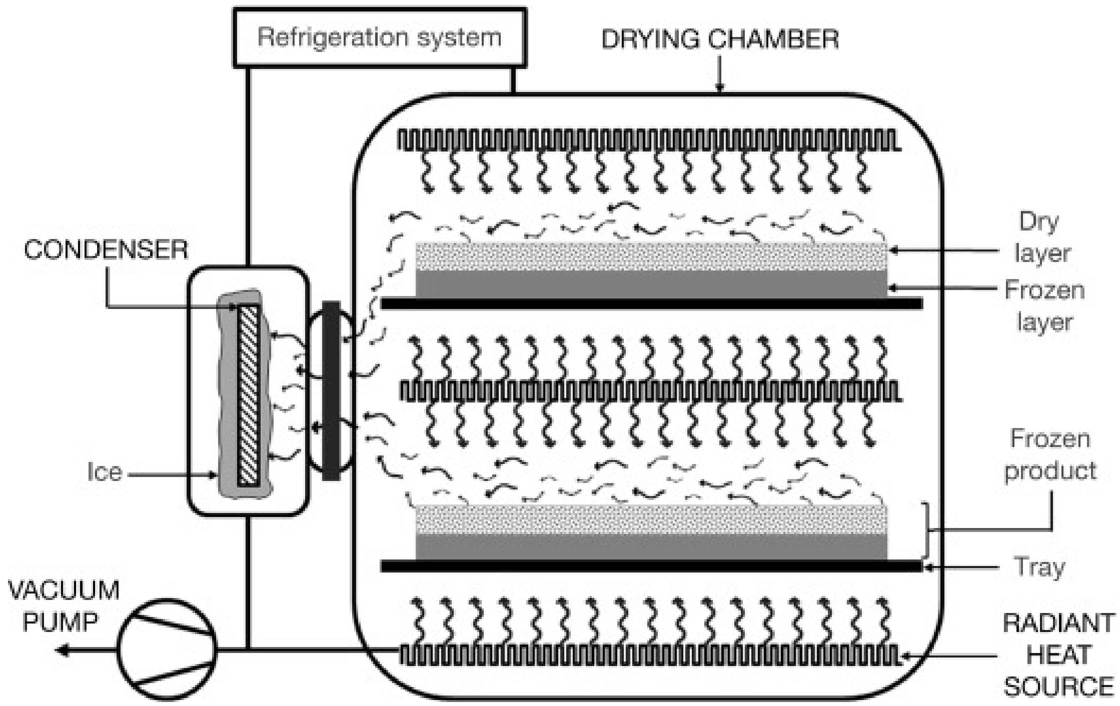


Figure 3-4: Schematic representation of freeze-drying (Garcia-Amezquita et al., 2016) **.**

3.3.3. Experimental Design and Optimization

In this study, a completely randomized design approach was followed. The randomization of the experimental runs was carried out through Design-Expert software version 11. A factorial design was chosen for planning the experiments and the evaluation of the experimental results was carried out by the Analysis of Variance (ANOVA).

3.3.4. Two-Level Factorial Design

The laboratory experiment was based on a factorial design where the different treatment factors (i.e. concentration of feed solution, drying temperature of freeze dryer) were analyzed for the different combinations of their test levels (feed solution concentration (25 %w/v to 33.3 %w/v) and drying temperature of the dryer (-50 to -30°C). Using CCD of response surface methodology (RSM), the optimum combination of the operational factors was determined. This is because of CCD features five levels for each factor and Provides excellent prediction capability near the center of the design space.Each of these two factors was evaluated for their effect at two levels and based on the result of the analysis, the optimum parameters were used to re-work the experiment for the validation of the optimum yield.

Table 3-1: Experimental Factors and Levels

Independent Variable	**Factor**	**Code levels**				
		-α	**-1**	**0**	**+1**	**+α**
Feed solution conc. (% w/v)	X_1	23.27	25	29.17	33.33	35.06
Drying temperature of dryer (°C)	X_2	-54.14	-50	-40	-30	-25.86

When these levels of factors were randomized using CCD, 13 runs were generated (Table 3-2).

Table 3-2: Randomized Experimental Runs

Std	Run	Factor 1: A-Feed conc. % (w/v)	Factor 2: B-Temperature (°C)
7	1	29.17	-54.14
11	2	29.17	-40
5	3	23.27	-40
2	4	33.33	-50
1	5	25	-50
10	6	29.17	-40
8	7	29.17	-25.86
9	8	29.17	-40
3	9	25	-30
13	10	29.17	-40
12	11	29.17	-40
4	12	33.33	-30
6	13	35.06	-40

3.3.5. Experimental Setup

3.3.5.1. Factors affecting freeze dried yield

Drying Temperature: Since temperature of the drier is one of the major factors that have a profound effect on physical and chemical properties of the freeze dried products,effect of drying temperature was studied by carrying out the drying process at different temperatures ranging from -50 °C to -30 °C low and high levels respectively for different gum Arabic solution concentrations (Lombrana and Villaran, 1997; Martinez-Navarrete, 2019; Maskat et al., 2014).

Concentration of aqueous solution: The concentration of the gum Arabic solution to be used for drying process has a significant effect on the accumulation of yield of powder recovery percent. This was tested by varying the concentration of gum Arabic within different solutions having concentrations from 25% (w/v) to 33.3% (w/v) low and high levels respectively for different drying temperature of the dryer (Saha et al., 2019).

3.3.6 Gum Arabic powder determination

3.3.6.1. Yield of powder

The percent of powder recovery is a measure of the concentration of aqueous solution efficiency to extract the powder from the prepared solution and it was defined as the amount of Freeze dried yield recovery. It is presented in percentage (%) and was determined for each experimental run (Mohammed and Roehle, 2011). The obtained precipitate was finally weighed to determine the yield (g) of dried gum Arabic powder. Eqt (3.1) was applied to determine the yield percentage of dried powder for each of the experimental runs.

% of Powder recovery = {(total weight of resulting powder)/ (feed solid content)}*100% (3.1)

3.3.6.2. Moisture content determination

The amount of moisture content for each of the powder yield was directly measured by using 1 g of the sample for about 1h in the MB45 moisture analyzer (Oikonomopoulou et al., 2011).

3.3.7. Freeze dried gum Arabic powder characterization

3.3.7.1. Fourier Transform Infrared Spectroscopy (FTIR)

The functional group analysis of the extracted gum Arabic powder was carried out using FTIR spectroscopy. A Perkin-Elmer spectrometer Spectrum 400 was used to obtain FTIR spectra. After the crystal area was cleaned, the solid material was placed on to the small crystal area; the pressure arm was positioned over the crystal sample area. Force was applied to the sample, pushing it on to the surface to form a transparent pellet that was analyzed using transmission method of 400-4000 cm^{-1} scanning range at a spectral resolution of 1 cm^{-1} and the spectrum was collected (Daoub et al., 2018).

3.3.7.2. Nuclear Magnetic Resonance spectrometer (NMR)

JEOL JNM-ECX500 spectrometer was used to obtain ^{1}H and ^{134}C NMR spectra. Deuterium oxide was used to dissolve Acacia gum samples (2% w/w) at 90 °C for 3hr before NMR analysis. ^1H and ^{13}C NMR spectra were recorded using a standard single-pulse sequence. Measurements were conducted at 50 °C (Marcone et al., 2013).

3.3.7.3. Thermo Gravimetric Analysis (TGA)

Nietzsche, TG 209 F3 Tarsus instrument that runs under Pro-teus Software on Windows operating system was used. About 13 mg of gum samples was weighed in sample pans and the weight loss against temperature from ambient to 400 °C at a heating rate of 10°C/min was recorded (Daoub et al., 2018).

3.3.7.4. Differential Scanning Calorimetric Thermo gram (DSC)

SDT Q600 V20.9 Build 20, Module DSC Standard Cell FC instrument, a Pan (T zero Aluminum Hermetic) and Nitrogen gas at flowrate: 50.0 ml/min. Sample of gum species (about 5 mg) was placed in a pre-weighed aluminum sample pan and the pan was sealed using a Quick Press pan crimper and the thermal data between 30 °C and 400 °C in nitrogen atmosphere with a heating rate of 10 °C/min were recorded. An empty pan served as the reference (Daoub et al., 2018; Martinez-Navarrete et al., 2019).

3.4. Statistical Data Analysis

The Design-Expert version 11 software was used for the statistical analysis. Generation of the model equation, main and interaction effects of the independent variables, and surface plots from the effects of the factors were evaluated for the gum Arabic powder yield. Based on their effect on the yield of the product, the factors were evaluated for their level of significance in the extraction process (Myers and Montgomery, 2002). A two-factor and two-level CCD design consisting of 13 runs for the total yield was employed, including five replicates at the center point. The experimental design was analyzed and done by Design-Expert version 11 software. The quality of fit of the regression model expressed as the coefficients of determination (R^2), the statistical significance determined by ANOVA and the response surface plots were all studied to estimate the models as well as to determine the optimum levels. For each factor, an experimental range is adjusted based on the results of the experiment data (Lundstedt et al., 1998).

CHAPTER FOUR

4. RESULTS AND DISCUSSION

The use of different types of freeze dryer parameters and variety of feedstock specificities has significant impact on the final product characteristics and the amount of yield. It is thus fundamental to realize how these variables influence the freeze drying mechanism in order to achieve an optimized operation (Silva-Espinoza et al., 2019). For instance setting the drying temperature is used as a basic parameter to get the desired type of freeze dried products (Nowak and Jakubczyk, 2020). Unless the right drying temperature is used, the final product becomes disqualified. Concentration of the prepared gum Arabic solution directly related to the amount of yield recovery. The processing variables (Concentration of feed solution (w/v %) and drying temperature (ºC)) were optimized using RSM to study their effect on the amount of yield, moisture content and the effect of independent variables on the prepared powder were checked (Duan et al., 2016; Oikonomopoulou et al., 2011).

4.1. Statistical Analysis

Table 4-1 summarizes the yield of gum Arabic powder and the amount of moisture content at each of the 13 experimental runs (Hammami and Rene, 1997). Evidently, a maximum yield of gum Arabic (214.1g) was obtained at the run number (4) and the least moisture content (6.07%) obtained at run number (7).

Table 4-1: Yield and moisture content at different processing conditions.

Std	Run	Factor 1: A-Feed conc. % (w/v)	Factor 2: B-Temperature (°C)	Response 1 Powder Yield g	Response 2 Moisture Content %
7	1	29.16	-54.14	202	12.8
11	2	29.16	-40	149.8	11.6

5	3	23.27	-40	120	9.05
2	4	33.33	-50	214	13.9
1	5	25	-50	160.3	11.1
10	6	29.16	-40	149.8	11.5
8	7	29.16	-25.85	97	6.07
9	8	29.16	-40	149.25	11.8
3	9	25	-30	96.35	6.63
13	10	29.16	-40	149.9	11.6
12	11	29.16	-40	150	11.3
4	12	33.33	-30	128.45	9.5
6	13	35.05	-40	180.13	13.1

As the above table 4-1 shows the amount yield of powder for gum Arabic varied within the range of 96.35g to 214.1g. The minimum powder yield was at -30 °C and 25% (w/v) concentration of gum Arabic solution at the run number (9) which was observed at the high level temperature and low level concentration of gum Arabic solution. On the other hand, the maximum powder yield was obtained at -50 °C and 33.3% (w/v) concentration of gum Arabic solution at the run number (4) which was observed at the low level temperature and high level concentration of gum Arabic solution (Patel et al., 2010; Harnkarnsujarit et al., 2012). In addition to this, the least moisture content was at -25.86 °C and 29.165%(w/v) concentration of gum Arabic solution at the run number (7) which was observed at the least level temperature and medium level concentration of gum Arabic solution. On the other hand, the maximum moisture content was obtained at -50°C and 33.3% (w/v) concentration of gum Arabic solution at the run number (4) which was observed at the high level temperature and high level concentration of gum Arabic solution (Krokida et al., 1998).

4.1.1. Experimental Design

4.1.1.1 Experimental Design for the Powder Yield

The experimental result was analyzed using Central Composite (CCD) of Design-Expert version 11 software to develop the model equation that can describe the significance of the extraction variables. The suggested choice of model was 2FI model that fits the data.

Table 4-2: Model summary for the powder yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	2.916E+05	1	2.916E+05			
Linear vs Mean	14748.08	2	7374.04	627.57	< 0.0001	
2FI vs Linear	**116.64**	**1**	**116.64**	**1218.49**	**< 0.0001**	**Suggested**
Quadratic vs 2FI	0.3218	2	0.1609	2.09	0.1947	
Cubic vs Quadratic	0.1996	2	0.0998	1.47	0.3152	Aliased
Residual	0.3401	5	0.0680			
Total	3.065E+05	13	23573.87			

Table 4-3: ANOVA for the Response of GA powder from feed solution

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	14864.72	3	4954.91	51761.92	<0.0001	significant
A-Solute Concentration	3648.15	1	3648.15	38110.71	<0.0001	
B-Shelf Temperature	11099.94	1	11099.94	1.160E+05	<0.0001	
AB	116.64	1	116.64	1218.49	<0.0001	
Residual	0.8615	9	0.0957			
Lack of Fit	0.5215	5	0.1043	1.23	0.4336	not significant
Pure Error	0.3400	4	0.0850			

The response of the analysis was dried gum Arabic powder yield. The response yield was arranged from 96.35g to 214.1g. The aim of the model fit summary was to maximize the adjusted R-Squared and predicted R-Squared values(Myers and Montgomery, 2002). Model significance was checked for the model and model factors. Linear model factors such as concentration of gum Arabic solution (A) and drying temperature (B) and, interaction term (AB) based on the F and p-values were checked.

Skimming along the last 2 columns of Table 4-3, it can easily be observed that the model is statistically significant for the p-value (<0.0001) is lower than 0.05. Values of " p-value" less than 0.05 indicate that model terms are significant. Hence, in this case, all model terms considered for this study (A, B, AB) are significant model terms. This shows that drying temperature, concentration of the feed solution, the interaction between drying temperature and feed solution concentration, affect the yield of the gum Arabic powder significantly(Myers and Montgomery, 2002; Myers and Montgomery, 2005).

The other important parameter that should be given attention here is the “lack of fit p-value” term, which shows the ANOVA statistics for unexplainable differences. This lack of fit p-value (0.4336) implies that lack of fit is not significant relative to the pure error. There is only a 43.36% chance that lack of fit p-value this large could occur due to noise.

Table 4-4: Fit statistics for the Response of GA powder from feed solution

Std. Dev.	0.3094	R^2	0.9999
Mean	149.77	Adjusted R^2	0.9999
C.V. %	0.2066	Predicted R^2	0.9999
		Adeq Precision	682.9423

The Predicted R^2 of 0.9999 is in reasonable agreement with the Adjusted R^2 of 0.9999 because the difference is less than 0.2.The "Pred R-Squared is the measure of the extent this developed model can be used to predict ranges of data this study has not considered, which, therefore, means that this model has 99.99% precision in fitting to overall ranges of data. The R-squared, on the other hand, shows how much of the difference in the outcome is explained by the model. This model is, therefore, almost is fairly good in accomplishing this task as demonstrated through its high value (99%). Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case, the ratio of 682.9423 indicates an adequate signal. Hence, it can be concluded that this model can be used to navigate the design space.

Table 4-5: Coefficients in Terms of Coded Significant Factors for the yield

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	149.77	1	0.0858	149.57	149.96	
A-Solute Concentration	21.35	1	0.1094	21.11	21.60	1.0000
B-Shelf Temperature	-37.25	1	0.1094	-37.50	-37.00	1.0000
AB	-5.40	1	0.1547	-5.75	-5.05	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the variance inflation factor (VIFs) are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable. In this case, one can simply observe that a good multi-collinearity exists for all the significant factors.

4.1.1.2. Experiment Design for the Moisture Content

As the table 4-6 below shows the suggested choice of model for moisture content response was quadratic model that fits the data.

Table 4-6: Model summary for the moisture content

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Mean vs Total	1506.62	1	1506.62			
Linear vs Mean	58.50	2	29.25	36.53	< 0.0001	
2FI vs Linear	0.0012	1	0.0012	0.0014	0.9712	
Quadratic vs 2FI	**7.82**	**2**	**3.91**	**146.86**	**< 0.0001**	**Suggested**
Cubic vs Quadratic	0.0528	2	0.0264	0.9895	0.4344	Aliased
Residual	0.1335	5	0.0267			
Total	1573.12	13	121.01			

The response of the analysis was moisture content. The response yield was arranged from 6.07% to 13.9%. The aim of the model fit summary was to maximize the adjusted R-Squared and predicted R-Squared values. Model significance was checked for the model and model factors. Model factors such as concentration of gum Arabic solution (A), drying temperature (B) and their quadratic terms $A^2$and B^2 based on the F and p-values were checked.

Table 4-7: Analysis of variance (ANOVA) for the Response of Moisture Content

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	66.32	5	13.26	498.24	<0.0001	significant
A-Solute Concentration	16.24	1	16.24	609.93	<0.0001	
B-Shelf Temperature	42.26	1	42.26	1587.49	<0.0001	
AB	0.0012	1	0.0012	0.0460	0.8363	
A^2	0.3862	1	0.3862	14.51	0.0066	
B^2	7.75	1	7.75	291.18	<0.0001	
Residual	0.1864	7	0.0266			
Lack of Fit	0.0544	3	0.0181	0.5491	0.6751	not significant
Pure Error	0.1320	4	0.0330			
Cor Total	66.51	12				

P-values less than 0.0500 indicate model terms are significant. From the above table it can easily be observed that the model is statistically significant for the p-value (<0.0001) is lower than 0.05. In this case A, B, A^2, B^2 are significant model terms. This shows that drying temperature, concentration of the feed solution, the quadratic drying temperature and quadratic feedstock concentration affect the moisture content significantly (Pikal et al., 1990; Pei et al., 2014).

The other important parameter that should be given attention here is the "lack of fit p-value" term, which shows the ANOVA statistics for unexplainable differences. The Lack of Fit F-value of 0.55 implies the Lack of Fit is not significant relative to the pure error. There is a 67.51% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good. The model fit summary statistics are listed in the table below.

Table 4-8: Fit statistics for the Response of moisture content

Std. Dev.	0.1632	R^2	0.9972
Mean	10.77	Adjusted R^2	0.9952
C.V. %	1.52	Predicted R^2	0.9911
		Adeq Precision	70.1512

The Predicted R^2 of 0.9911 is in reasonable agreement with the Adjusted R^2 of 0.9952 because the difference is less than 0.2.The "Pred R-Squared is the measure of the extent this developed model can be used to predict ranges of data this study has not considered, which, therefore, means that this model has 99.11% precision in fitting to overall ranges of data (Myers and Montgomery, 1995). The R-squared, on the other hand, shows how much of the difference in the outcome is explained by the model. This model is, therefore, almost is fairly good in accomplishing this task as demonstrated through its high value (99.72%). Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case, the ratio of 70.151 indicates an adequate signal. Hence, it can be concluded that this model can be used to navigate the design space.

Table 4-9: Coefficients in Terms of Coded Significant Factors for moisture content

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	11.56	1	0.0730	11.39	11.73	
A-Solute Concentration	1.42	1	0.0577	1.29	1.56	1.0000
B-Shelf Temperature	-2.30	1	0.0577	-2.43	-2.16	1.0000
AB	0.0175	1	0.0816	-0.1754	0.2104	1.0000
A^2	-0.2356	1	0.0619	-0.3819	-0.0893	1.02
B^2	-1.06	1	0.0619	-1.20	-0.9093	1.02

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.In this case, one can simply observe that a good multi-collinearity exists for all the significant factors.

4.1.2. Development of Regression Model Equation

4.1.2.1. Development of Regression Model Equation for powder yield

A linear model equation in terms of coded factors (equation 4.1) and actual factors (equation 4.2) shown below was fitted to the data model for predicting the response (powder yield). The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. In this case, therefore, only feed solution concentration (A) has an increasing effect on the amount of powder yield while drying temperature (B) and the interaction of feed solution concentration with drying temperature(AB) have a decreasing impact (Kortt, 1985). As implied by the largest positive coefficient in the equation, the highest positive impact on the yield went to the feed solution concentration, whereas the interaction of feed concentration and dying temperature had the highest negative impact.

$$\text{Powder Yield (g)} = 149.768 + 21.3546A - 37.2491B - 5.4AB \qquad (4.1)$$

The equation in terms of actual factors (equation 4.2) can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor (Shirley and Alistair, 1984). This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

$$\text{Powder Yield} = 2.48981 - 0.0589239 \times \text{Solute Concentration} + 0.0563865 \times \text{Inlet Temperature} - 0.129652 \times \text{Solute Concentration} \times \text{Inlet Temperature}$$

$$\text{Powder Yield} = 2.48981 - 0.0589239A + 0.0563865B - 0.129652AB \qquad (4.2)$$

4.1.2.2.Development of Regression Model Equation for moisture content

A quadratic model equation in terms of coded factors (equation 4.3) and actual factors (equation 4.4) shown below was fitted to the data model for predicting the response (moisture content). The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. In this case, therefore, feed solution concentration (A), interaction of feed solution concentration and drying temperature have an increasing effect on the amount of moisture content while drying temperature (B) and all quadratic terms have a decreasing impact (Lombrana and Villaran, 1997). As implied by the largest positive coefficient in the equation, the highest positive impact on the moisture content went to the feed solution concentration, whereas the quadratic terms of feed solution concentration had the highest negative impact.

$$\text{Moisture Content\%} = 11.56 + 1.4247A - 2.29846B + 0.0175AB - 0.235625A^2 - 1.05562B^2 \quad (4.3)$$

The equation in terms of actual factors (equation 4.4) can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

$$\text{Moisture Content \%} = -36.5438 + 1.15116 \times \text{Solute Concentration} - 1.0866 \times \text{Shelf Temperature} + 0.000420168 \times \text{Solute Concentration} \times \text{Shelf Temperature} - 0.0135829 \times \text{Solute Concentration}^2 - 0.0105562 \times \text{Shelf Temperature}^2$$

(4.4)

4.1.3.Model Adequacy Check

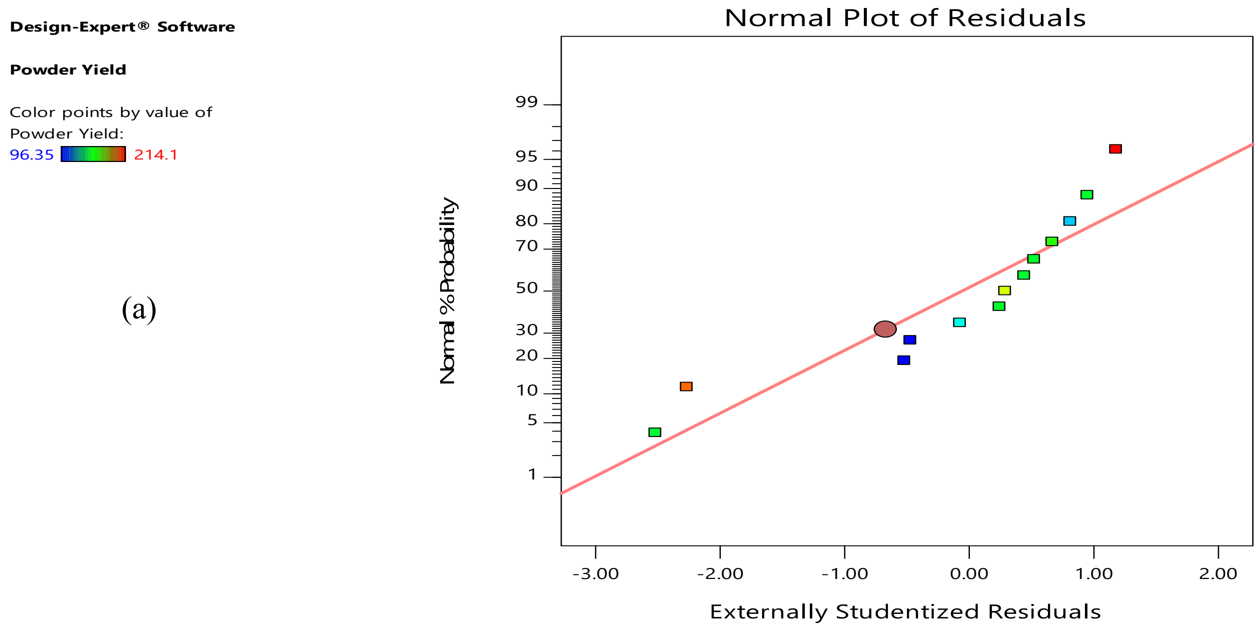
4.1.3.1.Model Adequacy Check for powder yield

It is always necessary to examine the fitted model to ensure that it provides an adequate approximation to the true system and verifies that none of the least square regression assumptions is violated. The quality of the model developed was, therefore, evaluated from their coefficients of correlation as summarized in Table 4-4 shown above. The coefficient of variance (CV), which is the ratio of the standard error of estimate to the mean value of the observed response, is a measure of reproducibility of the model. As a rule, a model can be considered reasonably reproducible if its CV is not greater than 10%. In this case, the CV is 0.2066% which means that the model is reasonably reproducible (Raza et al., 2019).

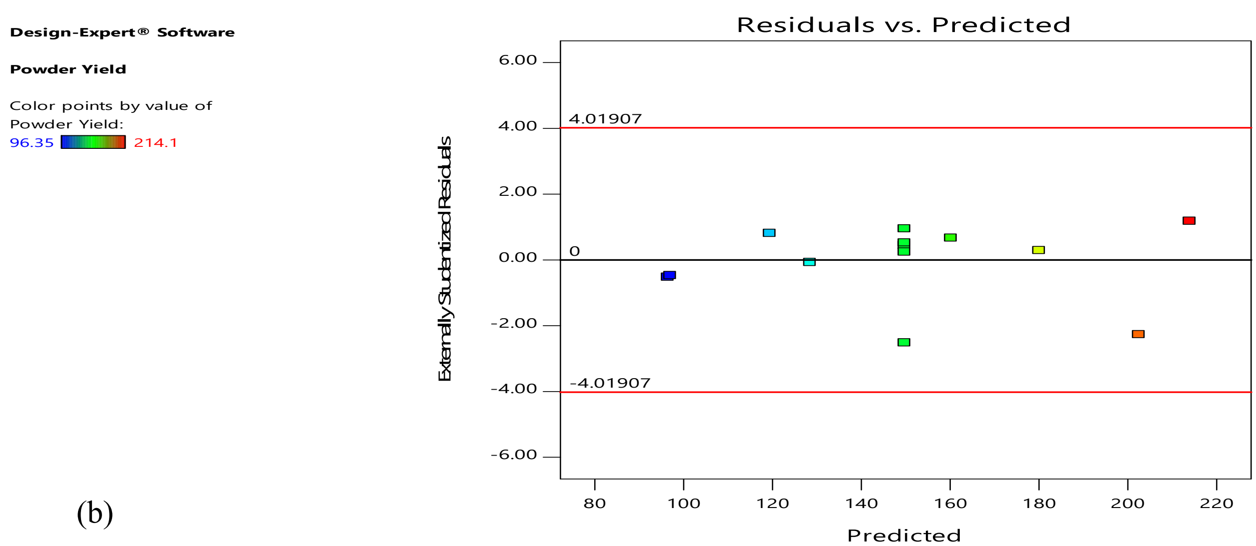
The regression model was found to be significant with the correlation coefficients of determination of R-Squared, adjusted R-Squared and predicted R-Squared which took the values 0.9999, 0.9999 and 0.9999 respectively. The highest percentage value of the R-squared term (99%) implies that there is no total variation in the powder yield which was not explained by the model. The “Adeq Precision" measures the signal to disturbance ratio due to random error. A ratio greater than 4 is desirable. In this case, as indicated by Table 4-4, the value is found to be 682.9423, which is a measure of the highly adequate signal.

The normal probability plot of residuals is shown in Figure 4-1 (a). If data from experiments form a normal distribution, the residuals fall on a straight line, which implies that the errors are spread in a normal distribution. Here a residual means difference in the observed value (obtained from the experiment) and the predicted value or fitted value (a value that can be predicted by the developed model equation). Hence, observing this normal plot, it can be noticed that all the points line up well and the deviation of points for powder yield from normality is insignificant, which means that the gum Arabic powder yield is precisely modeled.This can also be confirmed by the variations between the residuals and model predicted values analyzed through residual graphs as presented in Figure 4-1 (b). The plot is a random scatter (constant range of residuals across the graph). Thus, the model does not need any form of transformation as it is well fitted (AOAC, 2012).

On the other hand, Figure 4-1 (c) shows the plot of residuals versus the experimental run order. It checks for lurking variables that may have influenced the response during the experiment. The plot shows a random scatter and thus no more blocking or any other solutions were needed. The plot of predicted versus actual (Figure 4-1 (d)), which is the graph of the predicted response values versus the actual response values, is also another important diagnostics plot that should be taken into consideration. The purpose is to detect a value, or group of values, that are not easily predicted by the model. Looking into the graph, all values fall along the straight line, which shows the high power of prediction of the model developed.


Design-Expert® Software
Powder Yield
Color points by value of
Powder Yield:
96.35
214.1
Normal Plot of Residuals
Normal % Probability
99
95
90
80
70
50
30
20
10
5
1
-3.00
-2.00
-1.00
0.00
1.00
2.00
Externally Studentized Residuals

(a)


Design-Expert® Software
Powder Yield
Color points by value of
Powder Yield:
96.35
214.1
Residuals vs. Predicted
Externally Studentized Residuals
6.00
4.00
2.00
0.00
-2.00
-4.00
-6.00
4.01907
0
-4.01907
80
100
120
140
160
180
200
220
Predicted

(b)

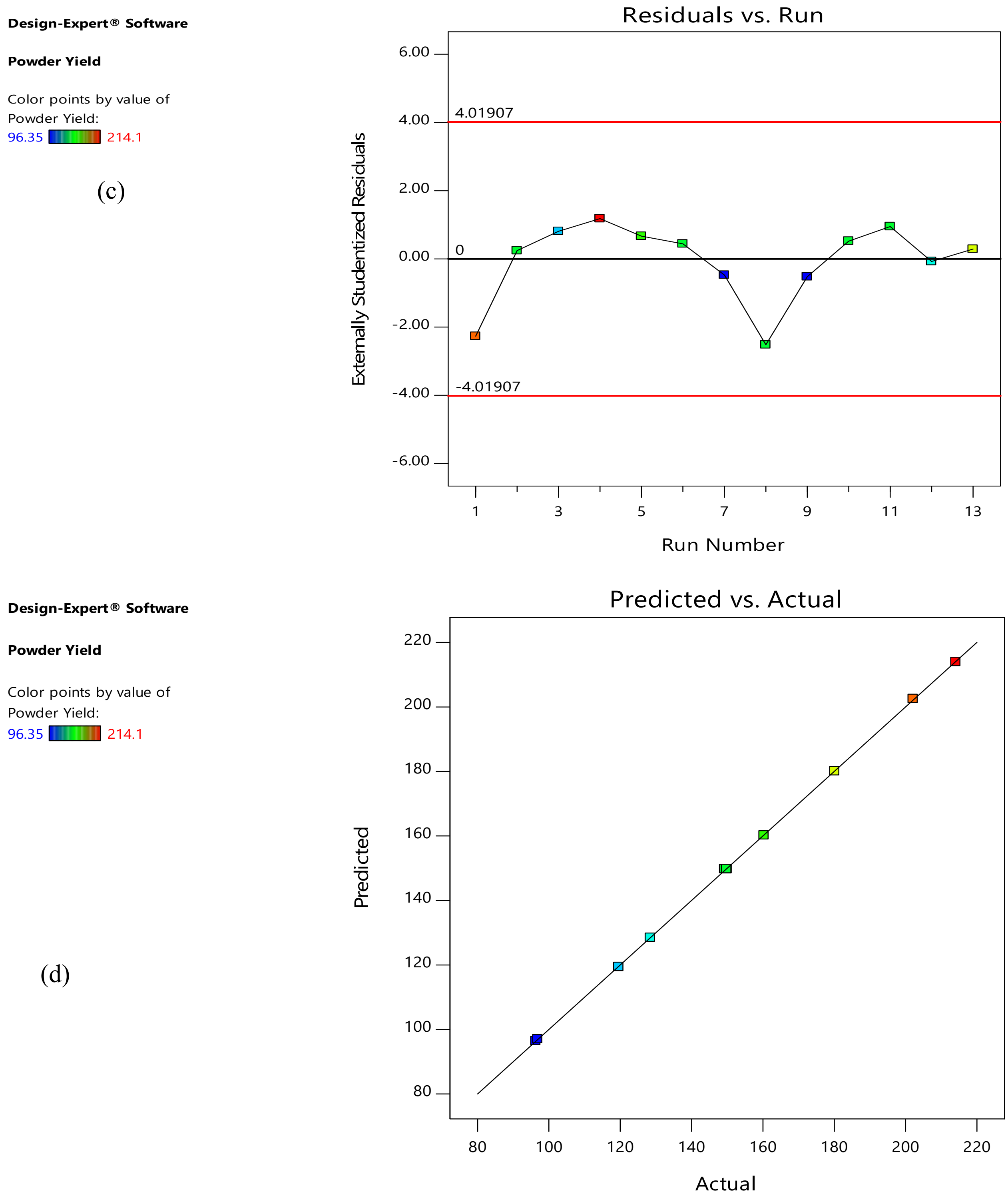


Figure 4-1: Normal plots of residuals yield (a); plot of residual versus predicted yield (b); plot of residuals against run order (c); plot of predicted versus actual (d)

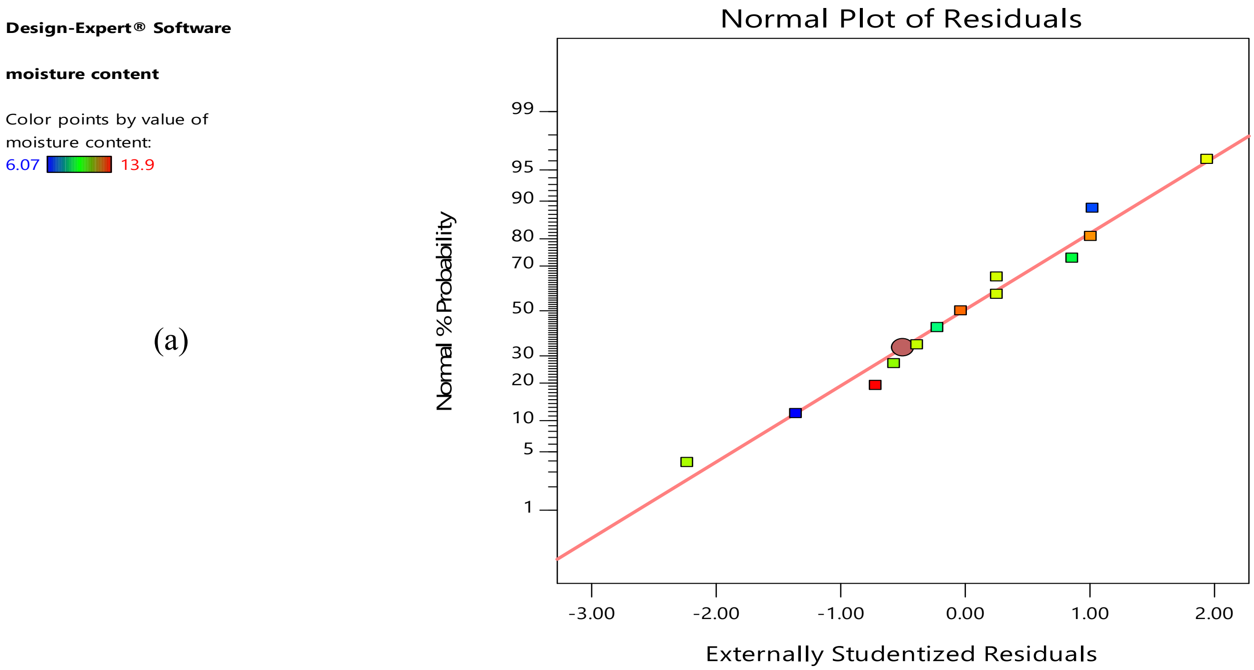
4.1.3.2.Model Adequacy Check for the moisture content

It is always necessary to examine the fitted model to ensure that it provides an adequate approximation to the true system and verifies that none of the least square regression assumptions is violated. The quality of the model developed was, therefore, evaluated from their coefficients of correlation as summarized in Table 4-8 shown above. The coefficient of variance (CV), which is the ratio of the standard error of estimate to the mean value of the observed response, is a measure of reproducibility of the model. As a rule, a model can be considered reasonably reproducible if its CV is not greater than 10%. In this case, the CV is 1.52% which means that the model is reasonably reproducible.

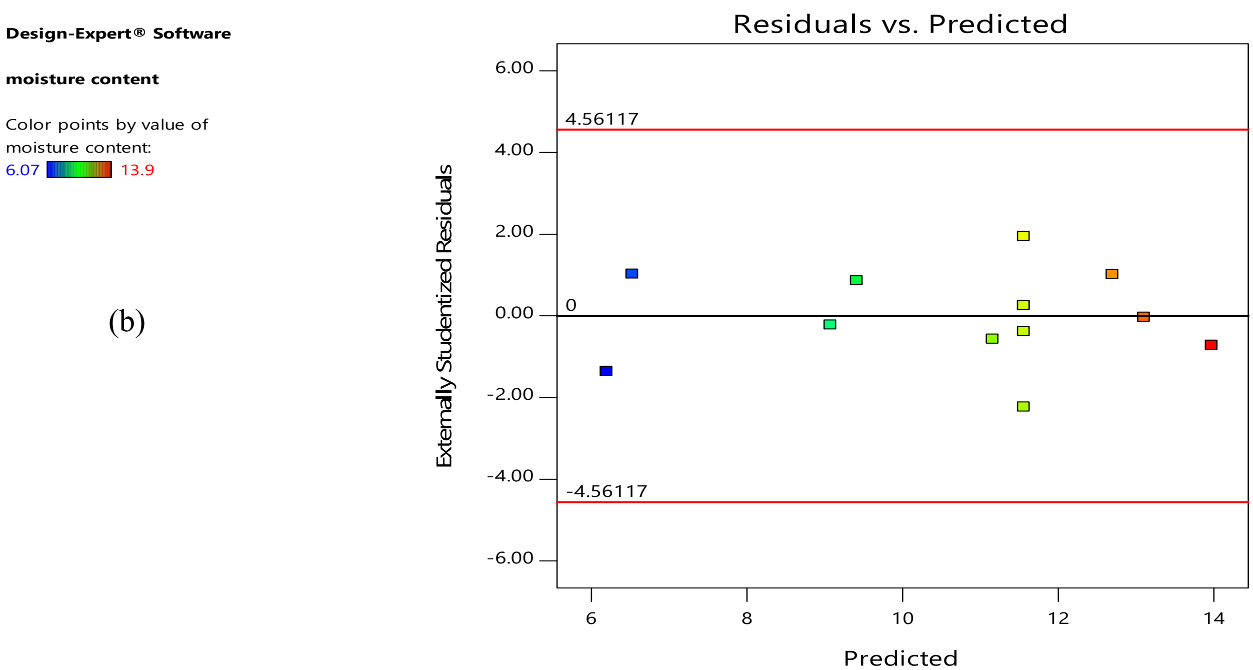
The regression model was found to be significant with the correlation coefficients of determination of R-Squared, adjusted R-Squared and predicted R-Squared which took the values 0.9972, 0.9952 and 0.9911 respectively. The highest percentage value of the R-squared term (99.72%) implies that there is only 0.28% of the total variation in the moisture content which was not explained by the model. The “Adeq Precision" measures the signal to disturbance ratio due to random error. A ratio greater than 4 is desirable. In this case, as indicated by Table 4-8, the value is found to be 70.1552, which is a measure of the highly adequate signal.

The normal probability plot of residuals is shown in Figure 4-2 (a). If data from experiments form a normal distribution, the residuals fall on a straight line, which implies that the errors are spread in a normal distribution. Here a residual means difference in the observed value (obtained from the experiment) and the predicted value or fitted value (a value that can be predicted by the developed model equation). Hence, observing this normal plot, it can be noticed that all the points line up well and the deviation of points for moisture content from normality is insignificant, which means that the moisture content is precisely modelled (Ekpong et al., 2016). This can also be confirmed by the variations between the residuals and model predicted values analyzed through residual graphs as presented in Figure 4-2 (b). The plot is a random scatter (constant range of residuals across the graph). Thus, the model does not need any form of transformation as it is well fitted.

On the other hand, Figure 4-2 (c) shows the plot of residuals versus the experimental run order. It checks for lurking variables that may have influenced the response during the experiment. The plot shows a random scatter and thus no more blocking or any other solutions were needed. The plot of predicted versus actual (Figure 4-2 (d)), which is the graph of the predicted response values versus the actual response values, is also another important diagnostics plot that should be taken into consideration. The purpose is to detect a value, or group of values, that are not easily predicted by the model. Looking into the graph, all values fall along the straight line, which shows the high moisture content of prediction of the model developed.



(a)



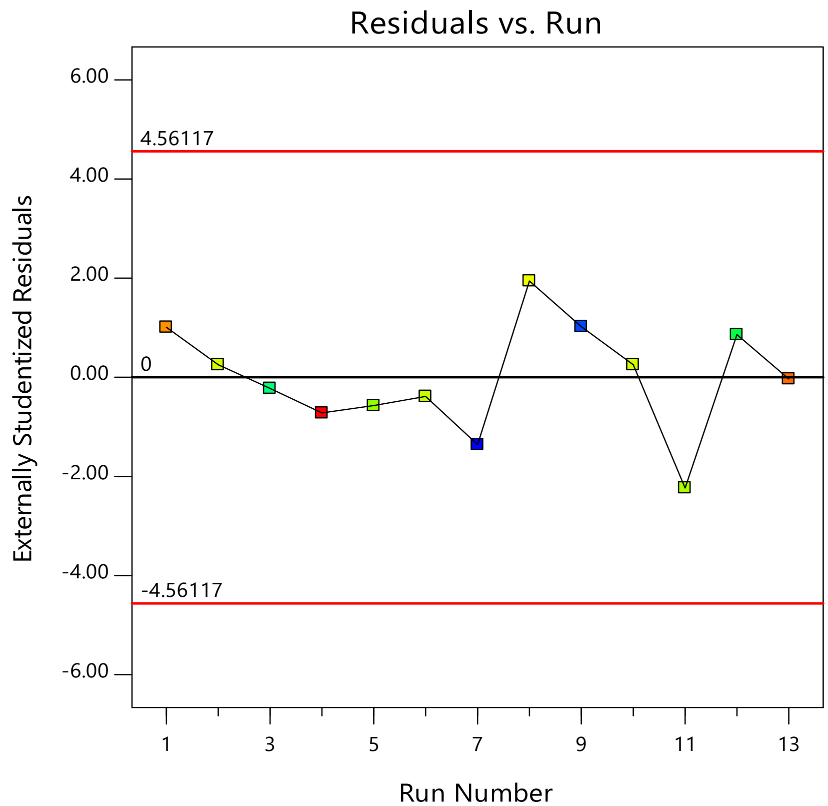
(b)

Design-Expert® Software

moisture content

Color points by value of
moisture content:
6.07 13.9

(c)



Design-Expert® Software

moisture content

Color points by value of
moisture content:
6.07 13.9

(d)

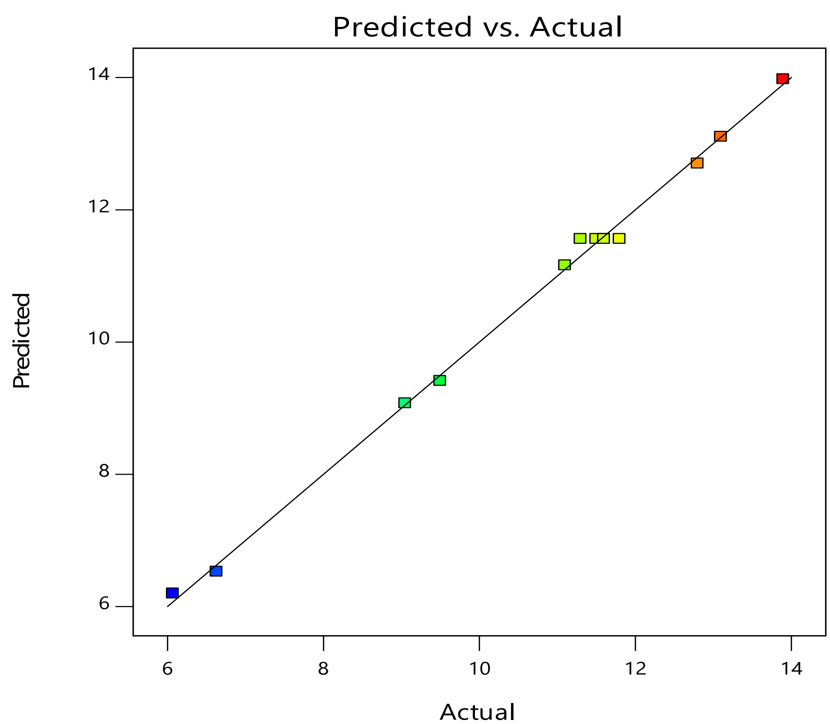


Figure 4-2: Normal plots of residuals moisture (a); plot of residual versus predicted moisture (b); plot of residuals against run order (c); plot of predicted versus actual (d)

4.1.4. Comparison of the Factor Effect

4.1.4.1. Comparison of the Factor Effect for the Powder Yield

Figure 4-3 below is called a perturbation plot, which helps to compare the effects of all the factors at a point in the design space. The response is plotted by changing only one factor over its range while holding all the other factors constant. A steep slope or curvature in a factor shows that the response is sensitive to that factor. A relatively flat line shows insensitivity to change in that factor. If there are more than two factors, the perturbation plot could be used to find those factors that most affect the response. In this case, as it can be seen from the plot, the increase in the feed solution concentration has an increasing effect on the powder yield while increasing the drying temperature decreases the amount of the yield which is reverted.

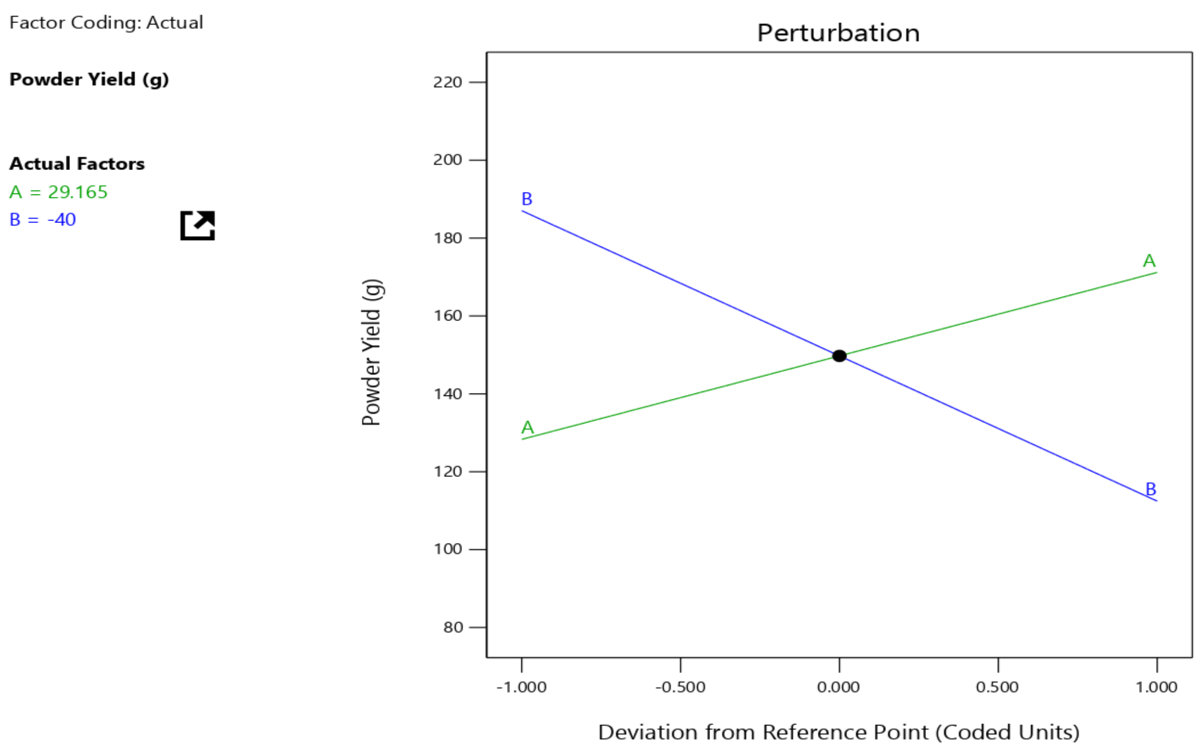


Figure 4-3: Perturbation plots comparing the effect of all factors at a point in the design space for powder yield.

4.1.4.2. Comparison of the Factor Effect for Moisture Content

Figure 4-4 below is called a perturbation plot, which helps to compare the effects of all the factors at a point in the design space. The response is plotted by changing only one factor over its range while holding all the other factors constant. A steep slope or curvature in a factor shows that the response is sensitive to that factor. A relatively flat line shows insensitivity to change in that factor. If there are more than two factors, the perturbation plot could be used to find those factors that most affect the response. In this case, as it can be seen from the plot, the increase in the feed concentration has an increasing effect on the moisture content while increasing the drying temperature decreases the amount of moisture content which is reverted(RP).

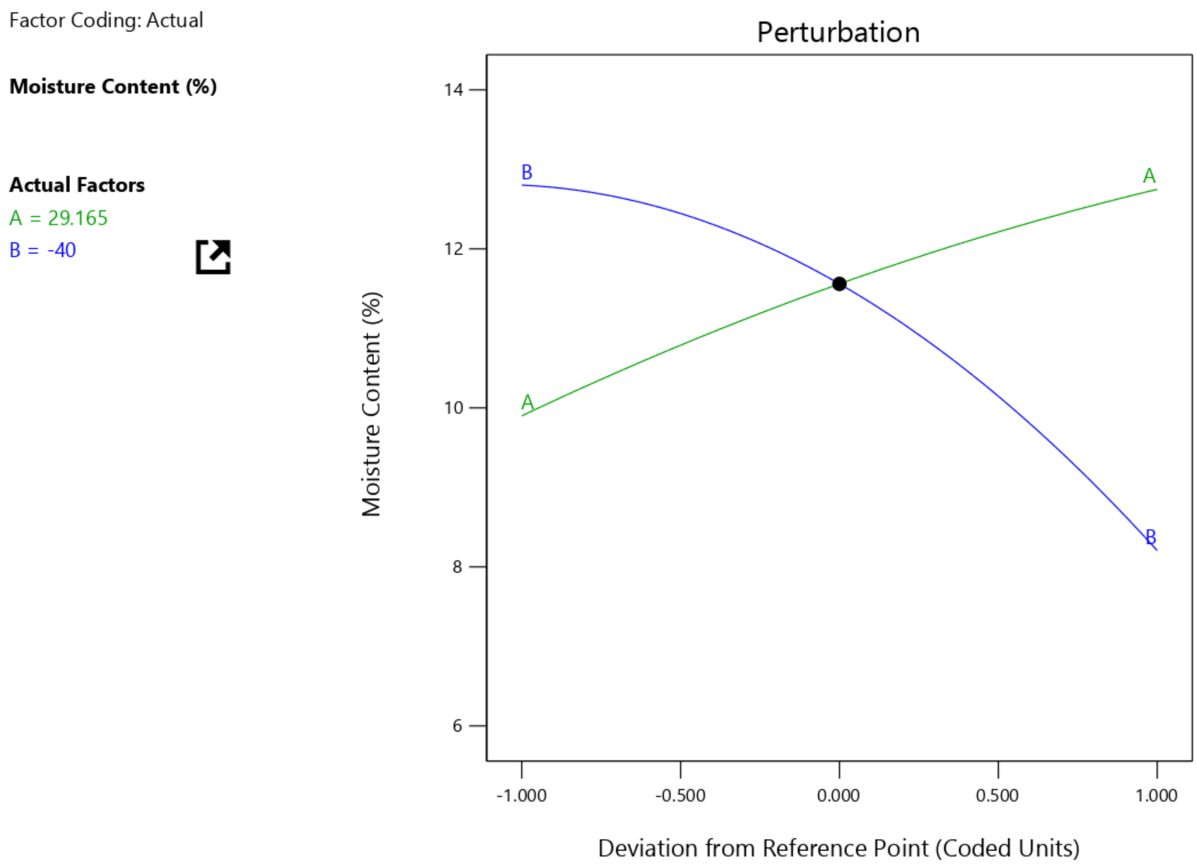


Figure 4-4: Perturbation plots comparing the effect of all factors at a point in the design space for moisture content

4.1.5. Effect of Production Process Variables

Based on the ANOVA result, both the powder yield and moisture content are significantly affected by two of the factors involved in this study. Below is the discussion for each of these factors and how they affect the two responses.

4.1.5.1. Effect of Temperature on the Yield of Gum Arabic

During drying process, setting the right drying temperature is one of the critical factors that have a profound effect on freeze dried products. In this study, a maximum dried gum Arabic yield was found at around -50 °C as illustrated Figure 4-3 below. Apparently, the higher temperature does not favor the yield of gum Arabic powder. In drying process, the lower temperature will not be strong enough to dry the moisture content of the feed solution.On the other hand, applying too high temperature which is beyond the right drying temperature, results disqualifying the desired product. Heating plate temperature also plays an important role in creation of the material structure during the freeze-drying (Tang et al., 2005). Undesired changes in the material structure can be a result of inappropriate temperature applied in the process. An excessive collapse of the structure may lead to a decrease of the freeze drying rate during secondary drying, as well as the deterioration of many features of products related to texture, porosity, volume, shape, stickiness, rehydration capacity, and stability. Hence, a wise choice of drying temperature, balanced on these factors, should be done according the feedstock properties. A lot of freeze dried foods have been produced by drying process; each of dried food has its own drying temperature. Therefor knowing the exact shelf temperature is fundamental point to get satisfactory dried yield (Edelmann 2002).

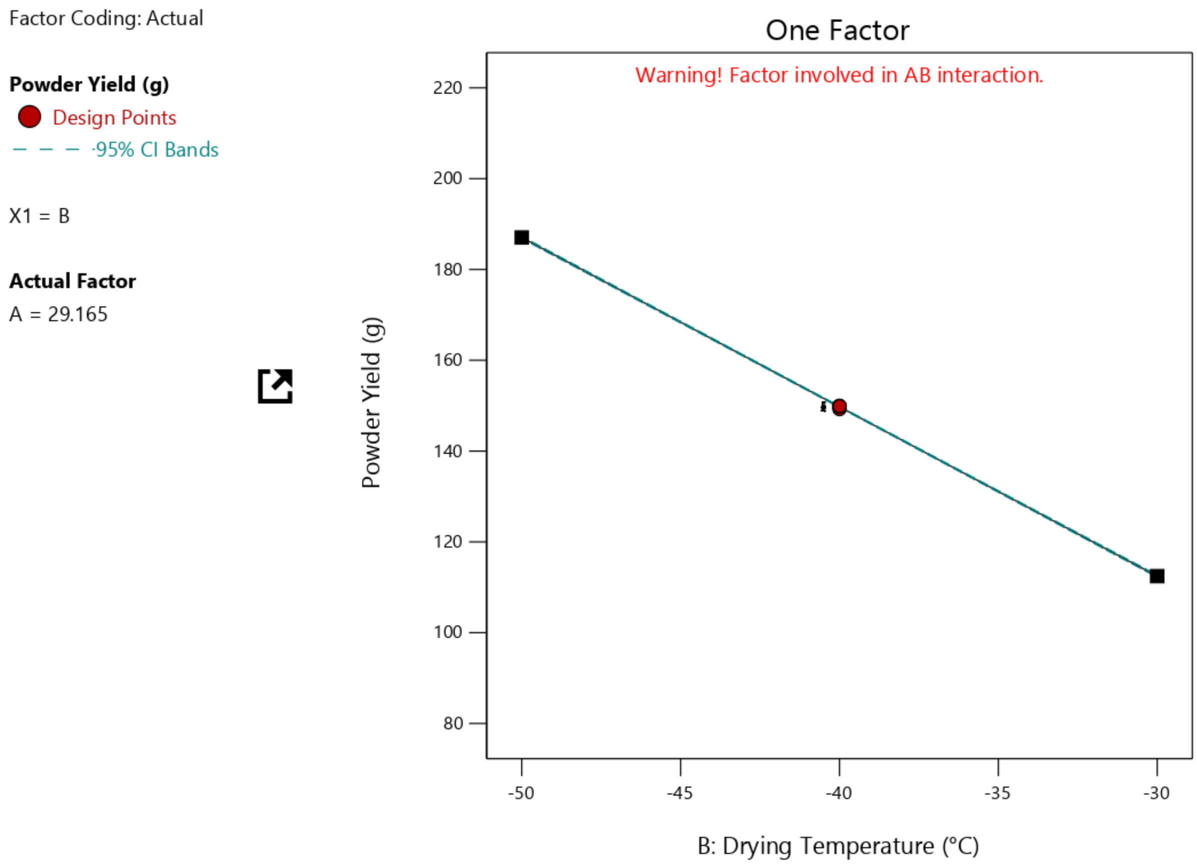


Figure 4-5: Effect of temperature on the powder yield

4.1.5.2. Effect of Concentration on the powder yield

The concentration of feed solution has significant effect on the amount of yield produced. It is related as directly proportionally. Which means increasing concentration increases the yield and also decreasing the concentration decreases the yield. As it can be seen the figure below, the highest powder yield is obtained on the concentration amount 33.33(%w/v). This shows that during spray drying process of gum Arabic powder, it is advised to prepare the higher concentration of feed solution to obtain the maximum yield as much as possible. But it is difficult to prepare gum Arabic solutions beyond 50%w/v since it becomes high viscous jelly like solution which is difficult to dry. In addition to this as the feed solution concentration increases, the viscosity also increases. Therefor this behavior of the feed solution makes not only drying process but also makes aqueous cleaning process to be the most difficult step starting from preparing the solution up to the last procedure (Oyinloye and Yoon, 2020).

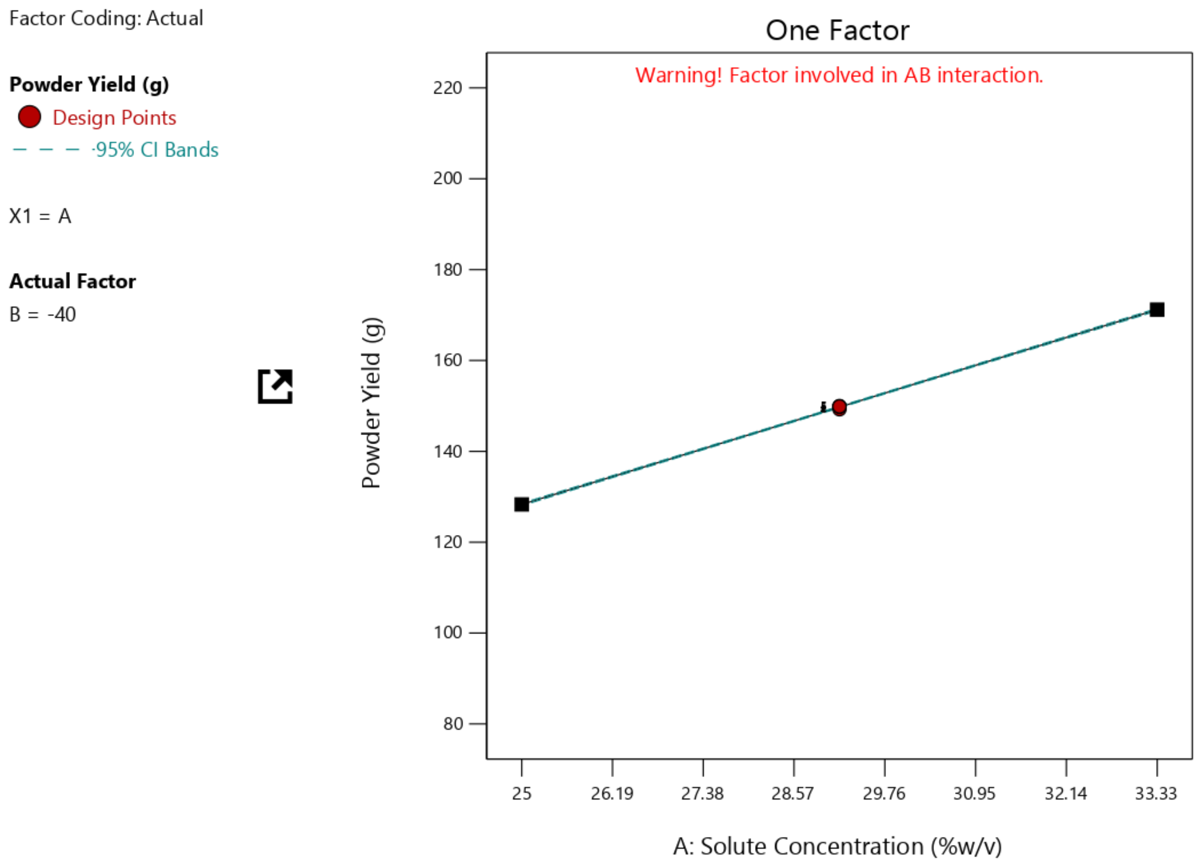


Figure 4-6: Effect of feed solution concentration on the yield

4.1.5.3. Effect of Temperature on the powder moisture content

It is known that the amount of moisture content is one of the most important qualities for dried products. Because moisture content is directly associated with the shelf life of the powder. So as much as possible, the experiments should be done with the parameters which result qualified product.one of the major factor which significantly influence the moisture content is the drying temperature. As presented in Figure 4-7, the amount of moisture content significantly decreases when the drying temperature increases. Therefor moisture content is inversely related with the amount of temperature. This is because the higher temperature provides high driving force which removes the amount moisture content. Because of this fact the least moisture content is obtained on the highest temperature amount as shown on the graph below. But further increasing of temperature may denature the product. Therefor based on the feedstock solution, drying process must be done with the right temperature range (Egas-Astudillo et al., 2020).

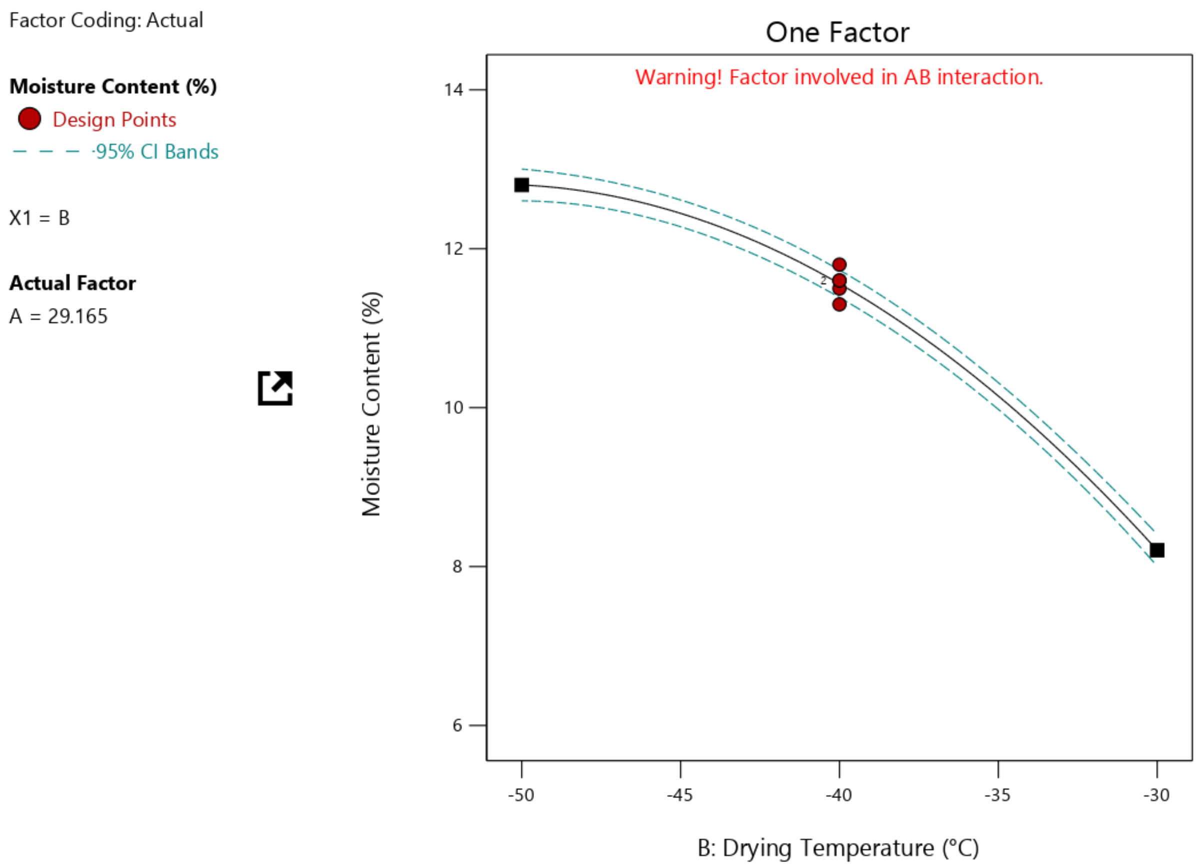


Figure 4-7: Effect of temperature on the moisture content

4.1.5.4. Effect of Concentration on the moisture content

As presented in Figure 4-8, the moisture content significantly increases with feed solution concentration.This means moisture content is directly related with concentration. This is because of the rising of feedstock concentration makes the solution to become viscousand this condition makes hard condition to dry the moisture content. This results the increasing of moisture content as the concentration increases. Therefore, increasing the concentration has increasing effect on the moisture content amount which disqualifies the powder quality, because high moisture content is undesired result in drying process (Anandharamakrishnan, 2017).

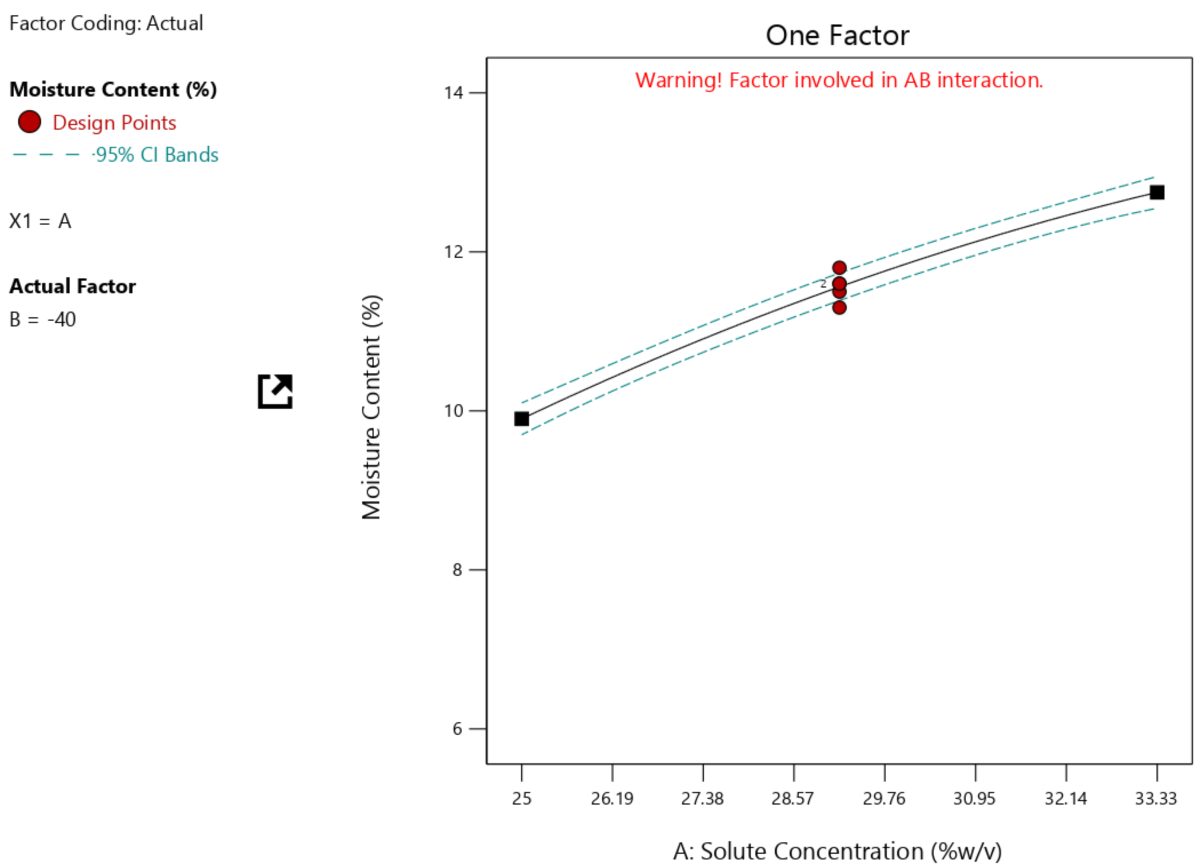


Figure 4-8: Effect of concentration on the moisture content

4.1.6. Effect of Interaction between Process Variables

The discussion of the single factors tells that each of the two factors has a significant effect on the response of powder yield and moisture content. However, it was also observed that there is an interaction between each of the variables (Table 4-3). For graphical interpretation of the interactions between regressor variables, use of 3D plots of the regression equation is recommended (Haseley and Oetjen, 2018). Interaction implies that the effect produced by changing one-factor level (for example, temperature) depends on the level of the other factor. In this research, to investigate the interactive effect of independent variables on the responses, three dimensional (3D) plots were examined to understand the interactions between two of the variables and the optimum levels of each variable for maximum yield of powder and minimum moisture content were determined (Adams et al., 2015). The interaction plots of drying temperature and feed solution concentration is shown in Figure 4-5 and Figure4-6 below.

4.1.6.1. Effect of Feed Concentration and Drying Temperature on Powder Yield

As discussed on the one-factor effect section, the same trend is also observed for the interaction effect of drying temperature and feedstock concentration. It is indicated that increasing the feed solution concentration as much as possible could result in a maximum result of gum Arabic powder amount about 214.1g.But increasing the drying temperature inversely related with the amount of yield which is the least amount 96.35g. When these two most important factors interact with each other, as indicated in Figure below, they could raise the powder yield to about 214.1 g, which means that the interaction effect of drying temperature and feedstock concentration is significant. This can be explained by the fact that during drying process, the high concentration increases the percent of powder recovery when there is proper selection of drying temperature for the feedstock solution (Ratti, 2001; Assegehegn et al., 2019).

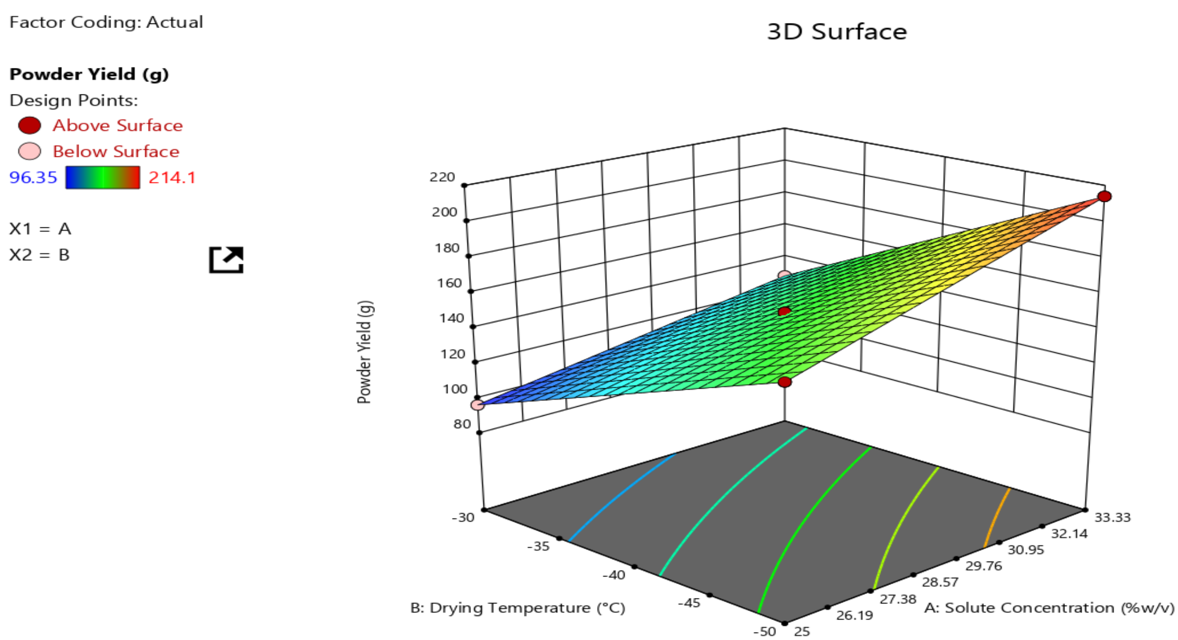


Figure 4-9: Surface plot of feedstock concentration and temperature interaction effect on powder yield

4.1.6.2. Effect of Feed Concentration and Drying Temperature on Moisture Content

As discussed on the one-factor effect section, the same trend is also observed for the interaction effect of drying temperature and feedstock concentration. It is indicated that the rise of feed solution concentration from 25%w/v to 33.33%w/v could result high moisture content amount from 6.07% to 13.9%. But the rise of drying temperature from-50C° to -30C° inversely related with the amount of moisture content which is the least amount 6.07% (Ekpong et al., 2016). When these two most important factors interact with each other, as indicated in Figure below, concentration could raise the moisture content while temperature decreases the moisture, which means that the interaction effect of drying temperature and feedstock concentration is significant. This can be explained by the fact that during drying process, the high temperature decreases the moisture content amount. Since having the least amount of moisture is one of the desired factors during drying process (Khalloufi and Ratti, 2003). The obtained result is significant.

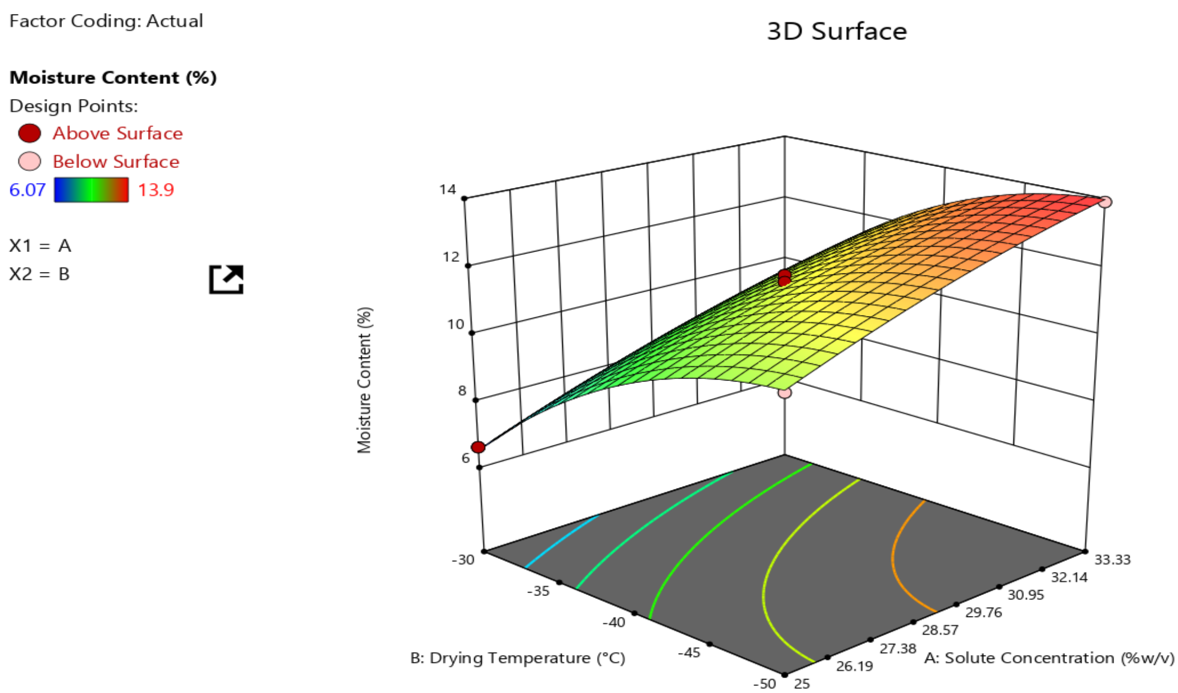


Figure 4-10: Surface plot of feed concentration and temperature interaction effect on the moisture content

4.2. Optimization of the Powder Production Factors

One of the objectives of the present study was to find the optimal process variables for the best gum Arabic powder yield. The process variables such as drying temperature and concentration of feed solution were optimized using the numerical optimization function of Design-Expert version 11 software. During optimization, drying temperature were set to be in range and concentrations were set to be maximized while yield of powder recovery was the response that needs to be maximized and the second response(moisture content) needs to be minimized (Lundstedt et al., 1998; Khalloufi and Ratti, 2003; Krzykowski et al., 2018). Table 4-5 shows the summary of factors, goals and the corresponding set of specific objectives that could optimize the process condition to have the highest response.

Table 4-10: Constraints Applied for Optimization

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Solute Concentration	maximize	25	33.33	1	1	3
B:Drying Temperature	is in range	-50	-30	1	1	3
Powder Yield	maximize	96.35	214.1	1	1	3
Moisture Content	minimize	6.07	13.9	1	1	3

The design expert under numerical optimization gave 9 different optimization solutions (which is a tabular form enclosed in the appendix section). Depending on the parameters by compromising yield, economy and energy carrying, the best solution was selected among the alternatives. Under taking the consideration of these constraints -31.7934 °C temperature and 33.33(%w/v) feedstock concentration were selected which gave a powder yield of 136.125g and moisture content of 10.1662% as summarized by Figure 4-11. Hence, for verification of these conditions, an experimental investigation was conducted at these optimum parameters then the powder yield was subjected to the analysis of FTIR, NMR spectrum, TGA analysis and DSC thermo gram (Daoub et al., 2018; Wu et al., 2019).

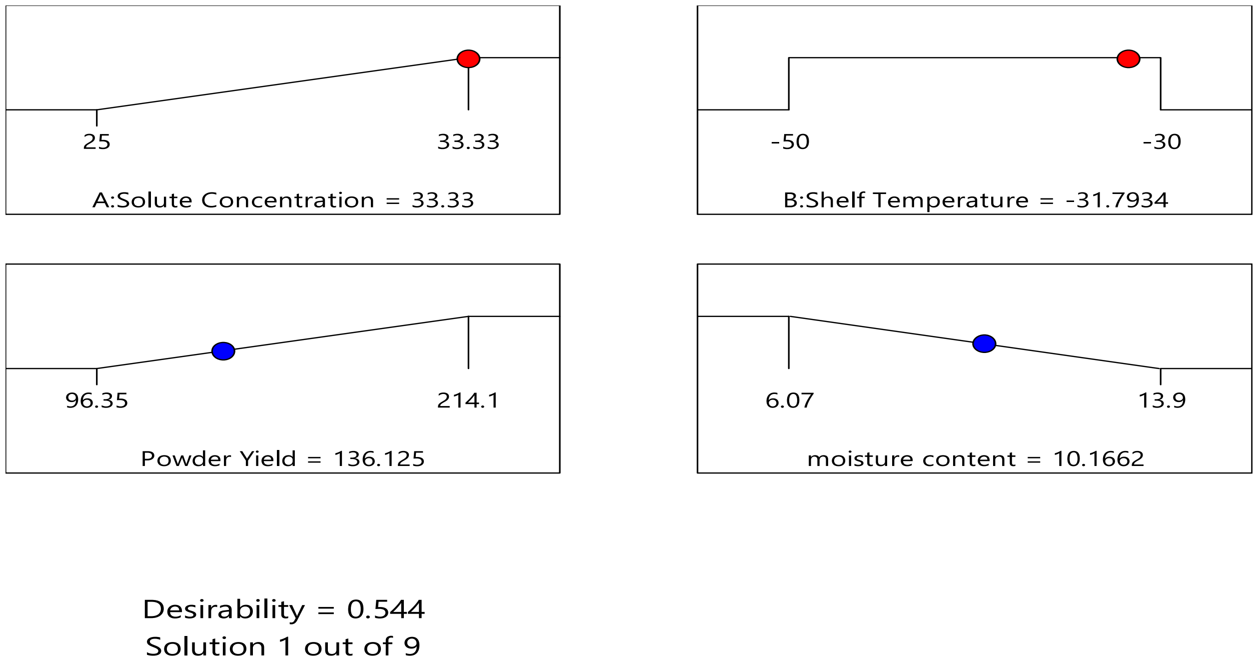


Figure 4-11: Ramp Plot of Optimization Solution for the Response

4.3. Product Characterization

4.3.1. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FT-IR) spectra of gum samples were recorded on an FTIR Perkin-Elmer Spectrophotometer was used at room temperature. The exudate gum samples were mixed with KBr and made into pellets. Standardization of instrument was carried out using KBr pellet as blank and the spectra were recorded in the range of 400-4000 cm^{-1}. Different organic functional groups present in Fourier transform infrared (FTIR) spectroscopy is recorded. The spectral bands falling between 3000–3600 cm^{-1} correspond to OH stretching modes. Peak observed at 3248.19 cm^{-1} in the spectra is therefore due to the presence of OH groups only. Peak present in the spectra at 1599.85 cm^{-1} correspond to the presence of COOH (carboxylic group). Bands identified in the spectra at 546.89 cm^{-1} is assigned to 1–6linkage of mannose (Eddy et al., 2014; Udo et al., 2017). FT-IR bands centered at 1029.84 cm^{-1} present in the FT-IR curve of gum arabic exudate is the result of arabinogalactan (Figure 4-12).

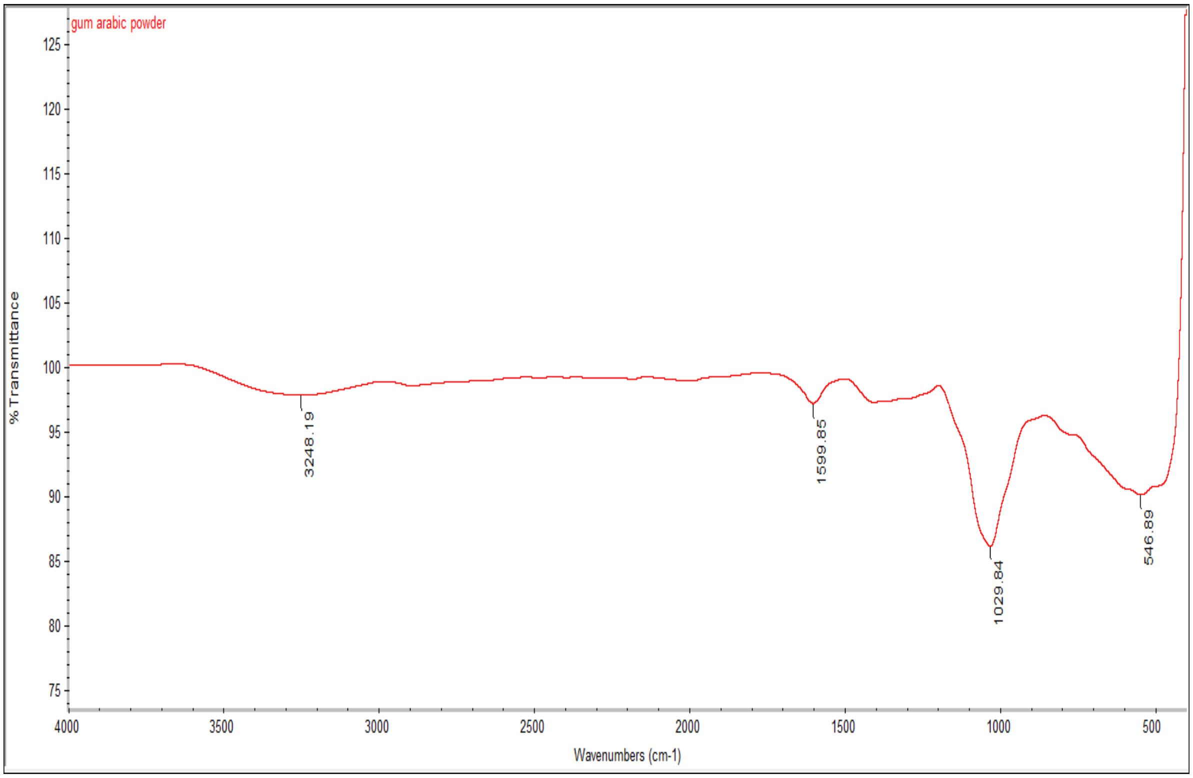
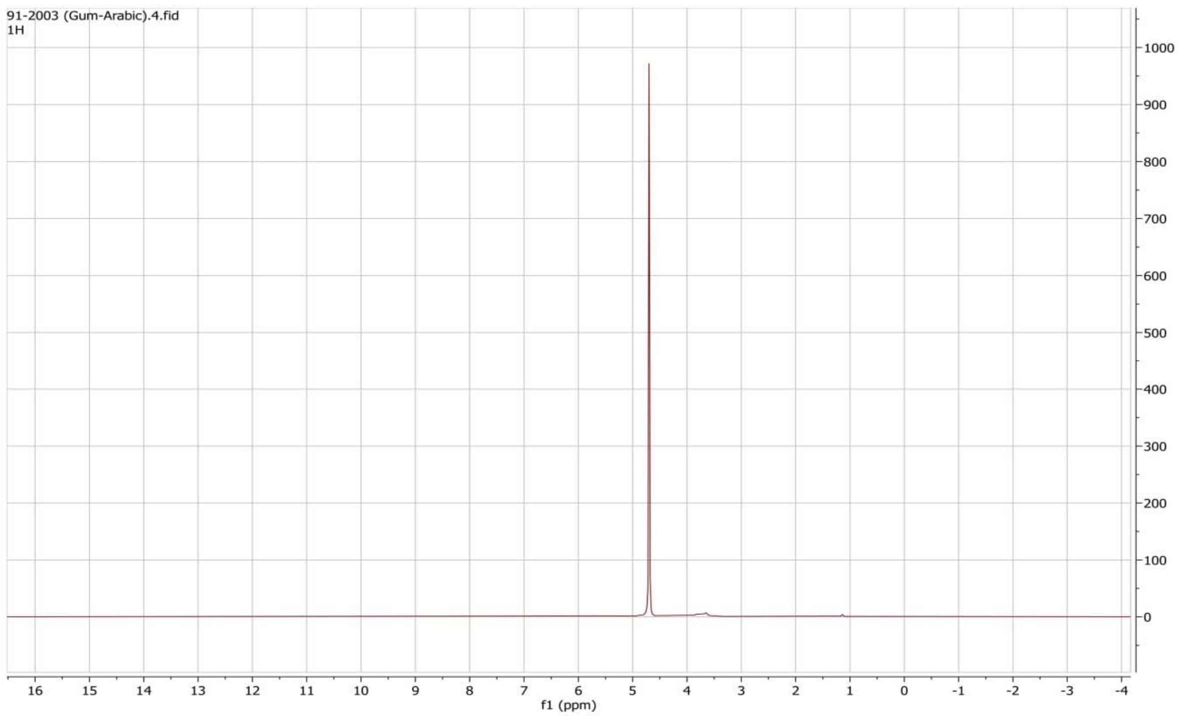
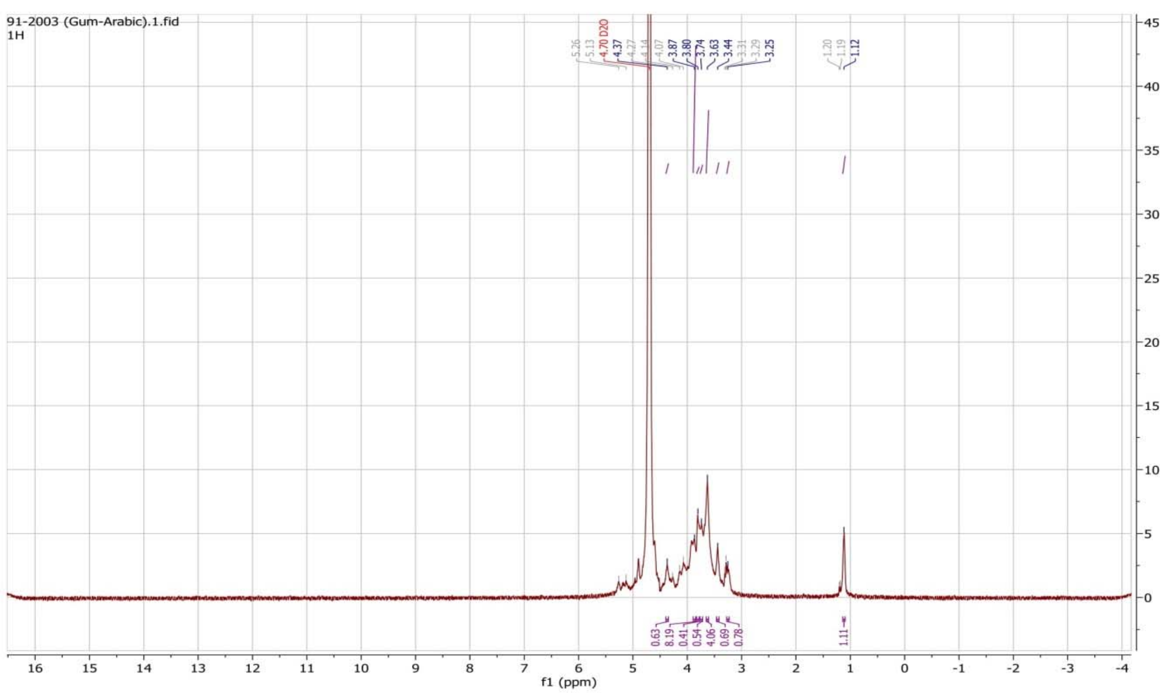


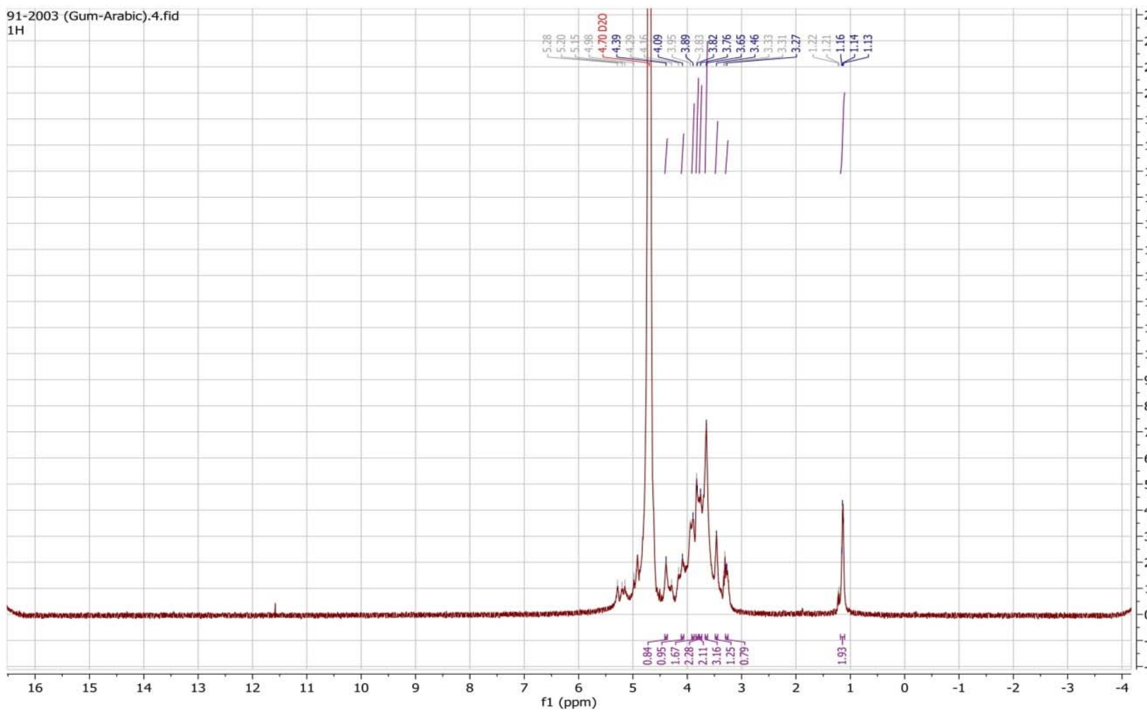
Figure 4-12: FTIR Spectra of Gum Arabic Powder

4.3.2. Nuclear Magnetic Resonance (NMR) Spectrometry

The ^{1}H NMR spectra, obtained at 55 °C, for AG is presented. Most of the chemical shifts of the protons from the saccharide ring was observed in the range between 3 and 4 ppm (3.65, 3.76, 3.82, 3.83). The anomeric protons appear between 4.5 and 5.5 ppm. The 'H NMR spectra of xanthan gum is measured usingJEOL JNM-ECX500 spectrometer. The peaks at 1.13, 1.14, 1.16, 1.21 and 1.22 are attributed to the pyruvate and acetate groups (Mulugeta et al., 2003). For the first time, quantitative yields are calculated from the integral of 'H signals corresponding to both substituents by reference to the equatorial anomeric proton, H-la, of mannopyranosic unit C, which is known as the more deshielded proton located at 4.09 ppm and 4.70 ppm (Andrikopoulos et al., 2003). In fact, 13C NMR cannot distinguish between substituents on units C and E; the position for substitution may be reversed (Figure 4-13).


91-2003 (Gum-Arabic).4.fid
1H
f1 (ppm)


91-2003 (Gum-Arabic).1.fid
1H
4.70 D2O
f1 (ppm)



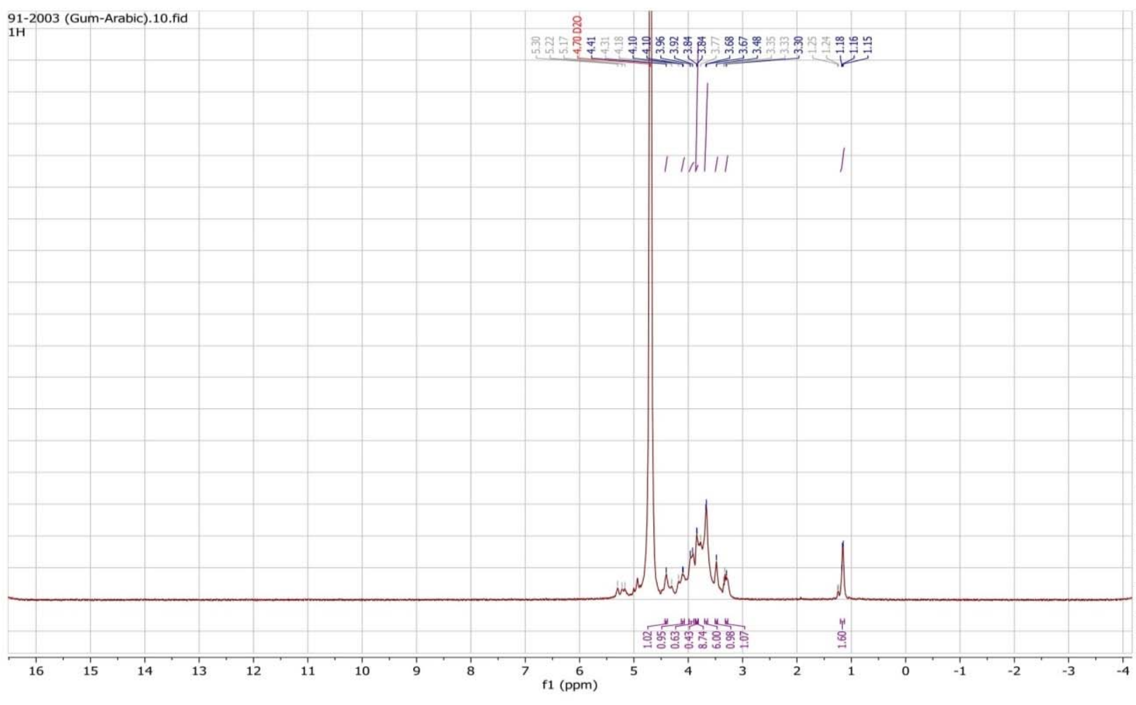


Figure 4-13: NMR Spectra of Gum Arabic Powder with different scanning rate

4.3.3. Thermo Gravimetric Analysis (TGA) with Differencial Scanning Calorimetry (DSC)

The thermogravimetric analysis is carried out using SDT Q600 V20.9 Build 20 instrument results are presented. As it is seen, there are three weight-loss stages. The first weight loss of sample occured in the temperature range 50 and 200 °C can be mainly attributed to the evaporation of water molecules bonded. This accounted for 10%. The second stage of weight loss is characterized by a dramatic loss of 60% in the temperature range of 225–350°C, was a result of dehydration of organic matter. This dehydration was coupled with an expected decarboxylation in the temperature range of 225-350°C. The final stage occurs in the 350–600°C range and is most likely due to complete decomposition of the glycoprotein of Acacia senegal. At temperatures exceeds 350°C, the biopolymer experiences unzipping and chain scission reactions which lead to a significant loss in molecular weight as well as thermal degradations (Alsadon, et al., 2023). The results clearly demonstrate the thermal stability of Acacia Senegal (Figure 4-14).

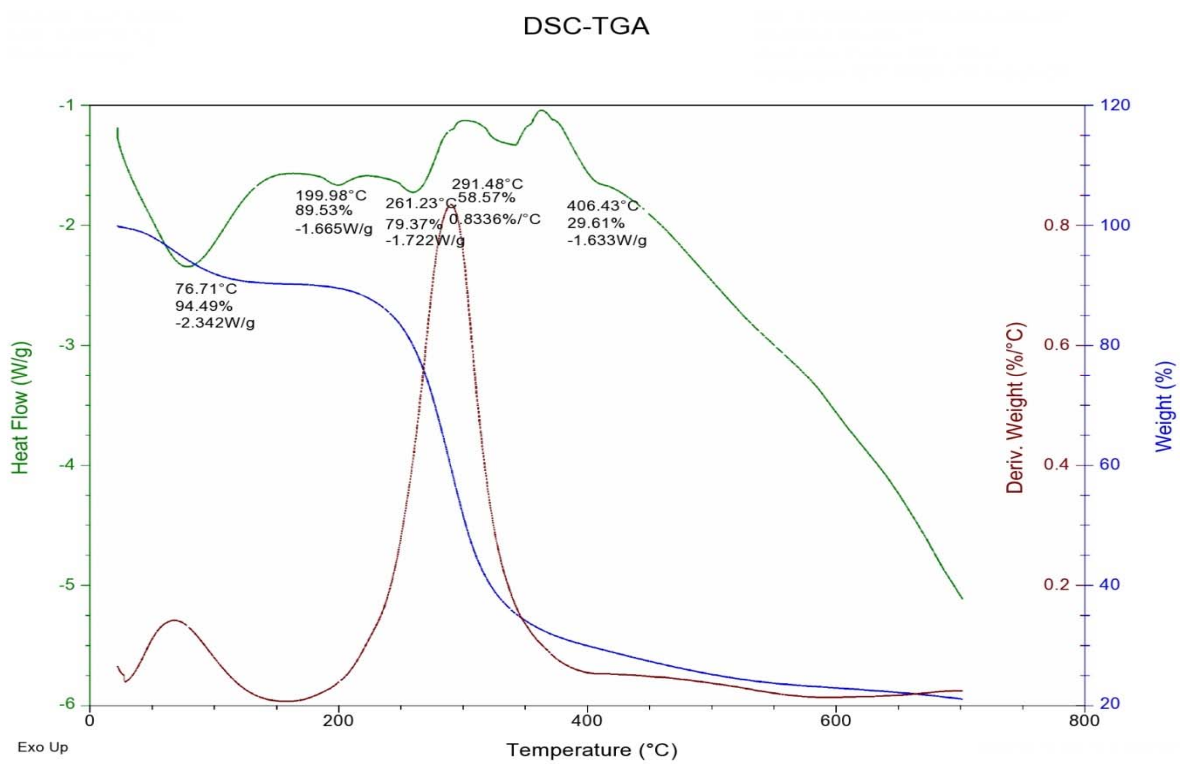


Figure 4-14: TGA analysis with DSC Thermogram for extracted acacia gum powder

CHAPTER FIVE

5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

It is known that acacia gum (gum Arabic) has numerous home and industrial applications. But naturally gum Arabic exudates are contaminated by sand, dirt, insects, pieces of bark and leaves, bacteria and so forth. To use this natural resource to different types of industrial application, finding gum Arabic purification mechanism is the first and the most important step. This research paper shows how to improve the quality of the product. In this study the main target focus on inventing the mechanism of producing pure instant freeze dried gum Arabic and developing optimization of the freeze drying operating conditions.

The experimental result showed that the maximum amount of yield (214.1g) was obtained at -50 °C and 33.33% (w/v) feed solution concentration while minimum amount of moisture content(%) was obtained at -25.86 °C and 29.165 % (w/v) feed solution concentration. RSM was applied to optimize the production factors and it was found out that all the main factors and their interaction could affect the drying process significantly where concentration has increasing effect on the two responses and temperature has a decreasing effect for the two responses.The optimum gum Arabic powder production with maximum yield 136.125g and minimum moisture content 10.16% was obtained at -31.79 °C temperature and 33.33% (w/v) concentration.

The obtained gum Arabic powder was tested for its character of FTIR, NMR Spectrum, TGA Analysis and DSC Thermo gram results.

Ethiopia is one of the countries located on gum Arabic belt in Africa. This shows there is wide availability of acacia gum in the country. Based on this research study, it is possible to make the instant acacia gum powder in this country. So the researcher encourages Ethiopian agricultural corporation to export such products rather than exporting the raw gum Arabic. This will have great financial income difference. This research can also help as a basis for other researchers to further study the drying process parameters which are not included in this research paper.

5.2. Recommendations

This research is concerned on processing acacia gum with optimization of drying parameters and characterization of the powder. The research is studied for the first time in our country. Therefore during the progress of undergoing this research paper, there were a lot of constraints. Based on this, the following recommendations were made and extensive researches are still needed.

- During drying process optimization, there are lots of parameters should be included in the input factors and the responses of the freeze drying process. But this research is limited only on temperature and concentration as input while yield and moisture content as response respectively. Therefor further investigations are also recommended on the development of optimization except those mentioned in this research.
- Encourage public awareness on the importance of acacia gum and its wide application should be studied.Therefore, the government must give especial attention to acacia gum processing technology through availability of processing machines and on the reduction of taxes to encourage others investors to invest in this sector.

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APPENDICES

Appendix A: Optimization possible results

Number	Solute Concentration	Shelf Temperature	Powder Yield	moisture content	Desirability	
1	**33.330**	**-31.789**	**136.105**	**10.164**	**0.544**	**Selected**
2	33.330	-31.932	136.717	10.222	0.544	
3	33.330	-31.637	135.456	10.103	0.544	
4	33.330	-31.560	135.130	10.072	0.544	
5	33.330	-32.092	137.402	10.285	0.544	
6	33.330	-31.444	134.634	10.025	0.544	
7	33.330	-31.325	134.124	9.976	0.544	
8	33.330	-31.192	133.556	9.921	0.544	
9	33.330	-30.827	131.994	9.768	0.543	
10	29.191	-50.000	187.208	12.812	0.378	

Appendix B: Images of Raw Material and Extraction Process

Figure B-1: Primary cleaned GA

Figure B-2: Prepared GA solution

Figure B-3: Filtration

Figure B-4: Centrifugation of GA solution

Figure B-5: Dusts removed

Figure B-6: Sterilization of GA solution

Figure B-7: Samples of freeze dryer flasks

Figure B-8: Moisture content analyzer

Figure B-9: GA powder sample

Figure B-10: Collection of raw material by Ethiopian agricultural coorporation