

Formulation and Characterization of Plant-Nutrient Tablets



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A thesis submitted in partial fulfillment of the requirements for the degree of
Master of Science in Plant Biotechnology

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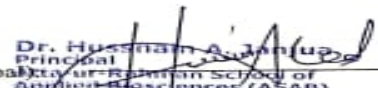
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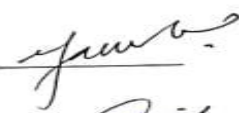
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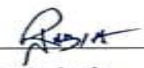
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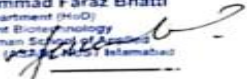
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I certify that this research work titled "*Formulation and Characterization of Plant-Nutrient Tablets*" is my own work. The work has not been presented elsewhere for assessment. The material that has been used in this work from other sources, has been properly acknowledged / referred.



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Dedication

“There have been times especially in recent past that I started feeling that I will not be able to move forward in life but every single time I got through the hurdles by the Grace of Allah. It’s all because of the blessings and constant support of my family and friends. I want to dedicate this thesis to my parents and my best friend because without their constant morale support, I would have not been able to come this far.”

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وَمَا تَوْفِيقِي إِلَّا بِاللَّهِ

"My Success is Only by Allah"

Durood-o-Salaam to our beloved Prophet **MUHAMMAD ﷺ** (PBUH), the last prophet of Allah and the prophet who had brought us from the darkness to the lightness.

اللَّهُمَّ صَلِّ عَلَى مُحَمَّدٍ وَعَلَى آلِ مُحَمَّدٍ
كَمَا صَلَّيْتَ عَلَى إِبْرَاهِيمَ وَعَلَى آلِ إِبْرَاهِيمَ
إِنَّكَ حَمِيدٌ مَجِيدٌ.
اللَّهُمَّ بَارِكْ عَلَى مُحَمَّدٍ، وَعَلَى آلِ مُحَمَّدٍ
كَمَا بَارَكْتَ عَلَى إِبْرَاهِيمَ وَعَلَى آلِ إِبْرَاهِيمَ
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Abbreviations

FT-IR	Fourier-transform infrared
XRD	X-ray diffraction
CRF	Control-release fertilizer
SRF	Slow-release fertilizer
RBD	Random Block Design
N	Newton

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Abstract

Densifying biochar loaded with nutrients can result in enhancing the conveyance and handling of the biochar. According to a hypothesis nutrient-loaded biochar tablets have the tendency for slowly releasing the nutrients embedded, in a synchronous manner. The objectives of this research study were to formulate the plant-nutrient tablets mainly consisting of biochar and characterization of the formulated plant-nutrient tablets. Different plant-nutrient tablets namely Copper-nutrient tablet, Boron-nutrient tablet, and manganese-nutrient tablet are formulated using the biochar powder and other needed additives which are essential in the process of tablet-making. Directly compressing the powder of biochar and salts (source of the nutrients) resulted in no formation of tablets, because of the low/or no binding capability. After the addition of binder i.e., 15% starch solution and filler tragacanth gum, tablets were formulated which were hard enough and fulfilled the basic criteria of the tablet making process. The best results for the harder tablets were achieved after performing multiple batches run with different combinations of the amounts of the ingredients on the single punch tableting machine. After making nutrient tablets for copper, boron, and manganese multiple tests were run on these plant-nutrient tablets keeping in view the prospects of the current study. FT-IR spectroscopy and XRD analysis confirmed the presence of the salts which were added as the source for the required nutrients i.e., copper sulphate pentahydrate, boric acid, and manganese sulphate monohydrate. Qualitative dissolution analysis shows that plant-nutrient tablets do not disintegrate into its components too fast and takes more than three months which is an important point in the current study while the hardness test confirms that plant-nutrient tablets formulated are hard enough that they can be transported after proper packaging. Prospects of this study include a long-term field study for carrying out the pot experiment and eventually confirming its findings

1. INTRODUCTION

1.1. Mineral Nutrition in Plants

Mineral nutrition's importance has been known to the world since ancient times. When ignition of plant at 400-600°C is done after being oven dried, organic materials' oxidation takes place which were present in the plant. Conversion of Carbon, Hydrogen, Sulphur, and Nitrogen takes place into CO₂, H₂O, SO₂, CH₄, NH₃, N₂ respectively and eventually liberates. Remaining incombustible matter in the end is known as plant ash. Plant ash's percentage varies from plant to plant. Forty elements in varying amounts are present in the ash. (Verma, 2008, p. 103).

All forty elements acquired after the plant ash's analysis are not considered essential for plants' nutrition except for the few which are needed for the development and growth of the plants and are considered essential. Elements which are not considered essential are called non-essential. C, O, and H are considered as elements which are essential for the plants since old times. Besides by sand-culture and water-culture experiments it is discovered that N, S, K, P, Fe, Mg, and Ca are other essential elements along with S, O, and H elements. Numerous disease symptoms can result from their deficiencies. B, Zn, Cu, Mn, Cl and Mo are other essential elements but are needed in smaller quantities in plants. C, O, H, N, P, S, K, Ca, Fe, Mg, B, Mn, Cu, Zn, Cl, and Mo are 16 essential elements known till date while others which are not needed by the plants for their development and growth are known as non-essential elements. (Verma, 2008, p. 103-104).

Essential elements can be bisected into two groups based upon how much amount does plants need. 1) *Macronutrients or Macroelements or Major elements*. Large quantities of these elements are needed (more than 100mg/l of water). C, O, H, N, P, S, Mg, K, Fe, Ca all these ten macronutrients play important role in body construction of the plant. 2) *Micronutrients or Microelements or Minor elements*. Smaller quantities of these elements are needed (100 or less

mg/l of water). Mn, Cu, B, Cl, Mo, and Zn all these six micronutrients play important role in various metabolisms. (Verma, 2008, p. 104).

Table 1: Differences between tracer and trace elements is shown which emphasizes upon importance of the nutrients which are required by the plants. (Verma, 2008, p. 104).

Trace elements	Tracer elements
Nutrient elements needed by the plants for various metabolisms and growth.	Radio isotopic elements needed for the detection of metabolic pathways.
Can be supplied to plants during deficiencies through nutrient solution.	Produced via radioactivity and are introduced into the plants through their different parts artificially.
Known as trace elements because they are needed in minute quantity for plant growth.	Geiger-Muller counter is used for their detection.
Major trace elements are Copper (Cu), Boron (B), Manganese (Mn), Zinc (Zn), Chloride (Cl), and Molybdenum (Mo).	Carbon (^{14}C), Sulphur (^{35}S), Nitrogen (^{13}N and ^{15}N), Cobalt (^{60}Co), Phosphorous (^{32}P), Magnesium (^{28}Mg), Potassium (^{42}K), Oxygen (^{18}O) etc.
Supplied through nutrient solutions (liquid medium).	They can be incubated in plants as liquid form or gaseous form. Deuterium and tritium, isotopes of hydrogen and nitrate ($^{15}\text{NO}_3$), etc., are given as water or in water but $^{14}\text{CO}_2$ is given in gas form.

1.2. Occurrence and Functions of Essential Elements

1.2.1. Manganese (Mn)

Mn in soil is found in form of bivalent ions, trivalent ions, or tetravalent ions. Of all these ions bivalent ions are only the one which are available in soluble form in soil solution. Mn can be found in plant ash especially in leaves. Manganese performs multiple functions like it participates in primary photochemical reaction of photosynthesis.(Arnon, 1971). Mn participates in chlorophyll formation.(Eyster et al., 1958). Indole 3-acetic acid (IAA) is also oxidized with the help of Mn. Various enzymes start working after they are activated by manganese which includes oxalosuccinate decarboxylase and malic dehydrogenase which are vital for Krebs cycle while hydroxylamine reductase and nitrate reductase enzymes which are important for nitrogen metabolism.(SPENCER et al., 1957). Manganese has another very important role i.e., working as a cofactor in oxidative phosphorylation.

Besides all these functions performed by this micronutrient, its deficiency can lead to many external as well as internal deficiencies to the plants. External deficiencies include elimination of the process of formation of the chlorophyll. Leaf necrosis and interveinal chlorosis. Growth and nitrogen assimilation is retarded. (Piper, 1941). Process of seed formation slows down.(Piper, 1941). Respiratory rate also lowers because ability of oxidase to carry oxygen is reduced due to manganese deficiency.

Internal deficiencies include chloroplasts losing chlorophyll.(Eltinge, 1941) and disintegration of starch grains e.g. in tomato.(Eltinge, 1941).

1.2.2. Boron (B)

B in soil is found in form of boric acid, silicates, calcium, and manganese borate. Its absorption takes place in form of tetraborate ions or borate anions. When compared with herbs, boron's percentage is greater in woody plants. Boron has multiple functions which includes helping in

the sugar's transport in phloem which eventually is used un numerous plant's metabolic activities. Changing vitamin's C concentration is also one function of boron. Besides helping in nitrogen, hormones, fats and phosphorous's metabolism, B also play part in the absorption of salts during the process of photosynthesis. (Verma, 2008, p. 115).

Boron deficiency can lead to many external as well as internal deficiencies to the plants. External deficiency symptoms include root apex and shoot's death, veins of the leaves turn into copper color, accumulation of amino acids and carbohydrates in the leaves, root shortening, retarded formation of root nodules in leguminous plants, "*top sickness*" disease in tobacco plant specifically. (Verma, 2008, p. 115).

Internal deficiency symptoms include causing browning and disintegration of the internal tissues in the sugar beet, a disease known as "*heart rot disease of sugar beet*", "*browning disease*", in cauliflower, cork cambium turns brown in apples, conducting tissues in tomato breaks down. Root cells' size is also reduced. (Verma, 2008, p. 116).

1.2.3. Copper (Cu)

Cu in soil is found in form of copper sulphide and chalcopyrite (CuFeS_2). Its absorption takes place in form of divalent copper cations. Copper has numerous functions e.g., formation of ascorbic acid oxidase and phenolase lactase enzymes, helps in chlorophyll's biosynthesis and CO_2 absorption during the process of photosynthesis, acting as a catalyst in the oxidation processes, imparting black pigmentation to *Aspergillus niger*'s spores, and acting as fungicide preventing diseases like "*late blight of potato*". (Verma, 2008, p. 116).

Deficiency symptoms caused include necrotic spots production at the tips of the young plants, production of the eruptions on the branches and the stems a disease called 'exanthema'.

Copper deficiency can also lead to 'Moor sickness' disease in leguminous plants, cereals and fruit trees. Here in spring plants' grow vigorously and size of the leaves is abnormally large but eventually they become chlorotic. (Verma, 2008, p. 116).

1.3. Importance of Fertilizers

It is estimated that in 2050 world's population will be increase by 2.3 billion. Although this rate of growth is slower when compared with the growth rate of last four decades but still it is important to bring it into account that with this boom in world's population, demand of the food will also rise. Keeping this in view fertilizers are needed in the agricultural sector. Nutrients are provided to plants by the fertilizers which eventually results in improved and increased crop yield.(Lawrencia et al., 2021). Fertilizers are one of the very important factors in the development of agricultural sector. Fertilizers are one important resource needed for swift increase in yields per acre.(*A Factor Analysis of Use of Fertilizers by Farmers.Pdf*, n.d.). With increasing world population, it is very important to double the production of the food grain too. Production of the crops can either be increased by virgin land's extension or increasing the crop yields. The fertilizers' role in increasing the production of the food grain is evident. Crops yields has been increased by more than fifty percent worldwide. Presently world's strategy related to fertilizers is increasing agricultural production by efficiently using fertilizers alongside with other inputs and also through improving the current farming methods(FAO, 1984)

1.4. Problems with the existing Fertilizers in Pakistan

Negative implications on the sustainability in longer run resulting due to the misuse of the fertilizers has been reported by several researchers. Misuse of fertilizers leads to environmental damage, degradation of soil increasing deforestation and depleting the base of natural resource. Efficient results are obtained when varieties used are fertilizer-responsive, preparation of land

is done precisely and other inputs such as water are provided timely. In Pakistan site-specific as well as general recommendations for the use of fertilizer are available but only few farmers take them seriously. The reasons for this negligence observed by the farmers are exogenous constraints like rainfall and internal constraints like limited availability of labor and cash. Institutions in Pakistan and fertilizer regulatory framework tends to overlook fertilizer practices' promotions which can result in improving the efficiency of the fertilizer use, e.g., urea's promotion is subsidized inspite of the fact that its use is reaching its optimal level, while nutrients like potassium and phosphorous are underutilized by the farmers as ignored by the policy of subsidy. Extension agents also place little effort in educating the farmers in reference to the practices which can improve the use efficiency of fertilizers like application methods, appropriate time of application and using different fertilizers in appropriate combinations. Use of fertilizer in a balanced manner is an important factor in improving its efficiency. Declining fertilizer and water partial productivities resulted in slowed growth in the total factor productivity (TFP), which later stagnated. TFP growth of Pakistan in 1980s was best around the world but eventually declined when compared with Asian competitors India, Sri Lanka, China. Inappropriate use of fertilizer can lead to inefficiencies at farm level as well as can cause degradation of the resources at a wider scale. Soil resources are also heavily damaged due to both fertilizer's under and overutilization as well as the poorly managed resources. Applications of the fertilizer nutrients even in developed countries have caused environmental contamination of soils and water supplies. Absence of the farming practices in Pakistan for adjusting the applications of the nutrients to land resources has caused several essential micronutrients of the soil to be over-mined like copper, iron, manganese, and boron. Content of organic matter has reached low levels due to the underutilization of the micronutrients.

1.5. Tablet

Tablets are most widely used due to their simplicity, low manufacturing cost and sophistication (Pawar, Rohit, *et al* 2014). The appealing qualities like color and texture are dependent on coating techniques (Gaikwad and Kshirsagar, 2020). The coating of a tablet basically depends on the purpose of the tablet. The present work aims to formulate a charcoal tablet that will burn longer as compared to pure charcoal, by using natural and organic binding agents and fillers.

1.6. Tablet formulation

The compaction process proceeds in a set consisting of two punches and a die attached on a single punch tablet machine and this whole procedure consists of 4 steps: 1) filling of the die with material, 2) press and compression, 3) release and decompression, and 4) tablet ejection (Sun 2011). It depends on the type of machine where whether both punches move towards each other, or one remains constant where the other applies pressure.

In the first step, the powder in the form of granules is filled in the die. In this stage, displacement of restrained air particles in the granulated powder takes place and the sponginess of the powder is reduced without any significant hostility of the punches. The decrease in sponginess makes the loose powder attain the shape of the container without any force exerted by the punches (Kuentz and Leuenberger, 2000). The second step is where the compression of powder in the die takes place. It is where the granules are pressed with the most pressure to give them a uniform shape. The third stage is where the pressure applied in second stage is reduced to null and it is the decompression stage. The tablets are ejected in the stage 4.

1.7. Tablet mechanical strength

The strongness of a tablet is contributed to the characteristics of granules such as shape, size and elasticity (Jain, 1999) as well as the applied conditions including compression pressure and the speed of punches (Hiestand, 1997). There are different factors that access the strength of a

tablet. The rigidity of a tablet that under how much pressure does a tablet deform. Rigidity can be considered illogical as it has a basically a contradiction since it has a different meaning in the field of mechanical engineering (Endicott, Lowenthal, and Gross 1961). However, various research has considered it significant in an up-to-date pharmaceutical work (Kesisoglou *et al.*, 2015, Tejedor *et al.*, 2015, Badawy *et al.*, 2012, Battu *et al.*, 2007).

1.8. Tablet components

Tablet consists of two major ingredients. The active ingredient and the excipients. Excipients are necessary material used to allow manufacturing of strong tablets (Wood, 1906). The active ingredient in our case is charcoal and *P. harmala* seed powder and the excipient consists of the binding agents and fillers.

1.9. Particle engineering by wet granulation

The formation of clusters is achieved by wet granulation where binding solution is mixed with powder (Iveson *et al.*, 2001). The resulting cluster must be dried till the moisture content is less than 5 percent. These clustered particles are usually of bigger size than required for tableting. This step is necessary for proper flow of material in the machine, to increase the speed of tableting, for the purpose of eliminating dust particles and to improve the appearance of the product (Iveson *et al.*, 2001; Mehta *et al.*, 2005; Litster and Ennis, 2004). Granulation is a complex step as it involves various factors and the interdependency of the factors adds towards further complication in understanding and scaling up the difficulty level (Iveson and Litster, 1998). Materials that chiefly experience plastic distortion such as Microcrystalline cellulose (MCC) is a type of material that faces plastic distortion when being granulated and it loses its properties essential for tableting (Shi, Feng and Sun, 2011; Badawy, 2006; Habib *et al.*, 1999). This significant forfeiture of capabilities being disrupted during granulation is known as over granulation (Shi, Feng and Sun, 2010).

In the case of dry granulation, it is also challenged with a comparable problem resulting in the loss of little or zero sensitivity to the enlargement of granules (Wu and Sun, 2007). The granules of plastic material tend to have smaller surface area and do not fracture under compression due to formation of bonds between their particles (Maganti and Celik, 1994). The brittle granules under compression break and produce greater surface area for bonding. This tendency of fragmentation can be effective if established in addressing the problem of over granulation.

Aim and Objectives

1. Formulation of the nutrient loaded tablets.
2. Characterization of the formulated tablets.

2. REVIEW OF LITERATURE

Population of the world is expected to touch 9.5 billion by year 2050.(Kareem et al., 2021). Degradation caused by heavy flooding, desertification and industrialization have resulted in the diminishing of the arable land and it is seen as a big threat to world's food security.(Jie et al., 2002). Due to all these issues food requirements are also expected to double globally by 2050.(Brown et al., 2009). For resolving such issues and to improve and modify our agricultural sector steps are taken which can help in making agricultural sector more sustainable and successful. Greater input of water, pesticides and fertilizers alongside new technologies have made immense impact in modern agriculture. The production of the crop has been increased significantly and has also eventually played its role in strengthening the economy.(He et al., 2015). Despite excelling in these modern technologies environment has been completely ignored, due to excessive use of pesticides and fertilizers' over-application has resulted in eutrophication, water toxicity, air pollution, groundwater pollution, degradation of soil quality and even changing the existing balance of the ecosystems. These factors has raised questions regarding the modern agriculture's sustainability.(Chen et al., 2018). Keeping in view the constant increase in the food demand, agricultural system has no other choice but to use fertilizers in larger quantities, even though it have numerous environmental impacts which are undesirable.(Azeem et al., 2014). Despite using conventional fertilizers in larger quantities, low utilization of nutrients by the crop is observed with subsequent loss into environment through denitrification, leaching and surface runoff.(Azeem et al., 2016). Due to loss of fertilizer in this manner nutrient availability to plants is diminished or deprived in some cases, process cost increases and environmental damage is also done.(Li et al., 2018). To overcome these challenges slow-release fertilizers (SRFs) and controlled-release fertilizers (CRFs), are being produced and used.(Trenkel, 2013). Studies have revealed that SRFs and CRFs can

contribute into the efficiency of nutrient use and reducing the environmental damage as it is eco-friendly with minimal pollution.(Roshanravan et al., 2015)(Tian et al., 2021). Biochar-based fertilizers have come to the limelight since last two decades because it has the ability of improving fertility of the soil, promoting growth of the plant and crop yield.(X. Liu et al., 2019). Biochar has received attention to a greater extent in the recent past and is regarded as a tool which can be used for the preservation of the environment.(Luis & Moncayo, n.d.). Pyrolysis of biomaterials is done under very low oxygen supply and resulting charred by-product is known as biochar.(Lehmann et al., 2011)(Vijayaraghavan, 2016). Biochar is greatly valued because it is eco-friendly and cost-efficient and can be produced on large scale. Using biochar is advantageous in numerous ways like clean-up of soil, production of energy, mitigating greenhouse gases, waste management, improving nutrient holding and soil's water retention potential.(Laird, 2008)(Vijayaraghavan, 2021). Transforming biological materials into biochar helps in producing renewable energy like bio-oil and synthetic gas besides this, this transformation also contributes in decreasing the atmosphere's CO₂ content.(America et al., 2007). Structure as well as properties of soil are also enhanced when it is supplemented with biochar, like, pH of the soil, organic ingredients, water retention potential, cation exchange capacity and aeration conditions.(Atkinson et al., 2010)(Vijayaraghavan & Raja, 2014)(Warnock et al., 2007). Studies has also revealed that greenhouse gases' discharges and leaching losses of phosphorous and nitrogen in soil decreases significantly with the help of biochar.(Amutio et al., 2013). Biochar also has some additional properties which makes biochar very special like specific surface area is high, efficient functional sites which includes hydroxyl, phenolic, carboxyl, carbonyl, at its surface. Besides, mineral ingredients and distinct porous structure of biochar makes it a great sorbent for contaminants that may be present.(Pan et al., 2014). Characteristics of biochar mentioned above have a considerably impact the bioavailability, conversion and movement of the pollutants in soil.(Tan et al., 2015).

Plant is grown and put on sale in containers, millions of plants are grown in pots by the nurserymen. These plants are grown in soil mixture which is disease free as well as sterile. None of the soil mixture used contains adequate amount of food needed by the plants for their growth so what nurserymen do, they take additional steps to prevent the plants from dying by applying liquid fertilizers by watering periodically, incorporating the soil mix with chemical fertilizers or applying granular fertilizers in measured amounts periodically. Usually, frequent supply of fertilizers is needed because fertilizer's nutrient value is used up very quickly, moreover, larger proportion of fertilizer's nutrients is lost due to leaching. When plants are delivered to a nursery by the wholesale nurserymen, it is desired that every plant's container should contain enough fertilizer, which can be used for several months by the plants or at least till the plant is sold further. Plants bought from nursery should have containers with sufficient supply of fertilizers is also desirable because the people who bought plants neither always place them into the ground nor fertilize them either. So if plants' containers are already filled with enough fertilizer through which plants can get sufficient nutrients for many months then it will be very helpful for the purchasers as now they only need to water the plants and that's it.(Heilig, 1994). The idea is to provide the plants with the nutrient tablet that can help the plant in overcoming the deficiencies caused due to insufficient amount of micro and macro-nutrients. Fertilizers in tableted form has been used in past but desired results have not been accomplished. Some tablets were incorporated with soluble fertilizers which plants used up very rapidly or even before being utilized by the plants they were leached out. In contrast to this some tablet fertilizers formulated were used when planting trees into the ground were used, which solubilizes at a very slow rate and those tablets were regarded as non-disintegrating tablets because they lasted for longer periods of time like for more than two years. No dispersal or distribution was required when applying these tablets because trees are planted into the soil and these fertilizer tablets are held onto their location by the soil.(Heilig, 1994). These

fertilizers tablets were not suitable for the container use in nurseries because these tablets neither provide enough food in the beginning nor make it available for the plants swiftly enough for the plants which are grown in the containers as these tablets are hard and are not broken for longer time intervals hence do not allowing soil moisture to make it solubilize and not providing enough surface face area for the bacterial activity. Bacterial activity is very important because many ingredients are insoluble in the absence of bacterial activity and without the needed activity of bacteria these tablets take too long for being broken and release the required nutrients. A nutrient tablet is needed which should contain necessary nutrients for the development and growth of the plant and keep providing them slowly for several months. A tablet is required which upon watering should slowly break down and start releasing required nutrients and providing them to plants in their available form after converting them from their unavailable form. Conventional fertilizers which are currently used can also create salinity problem which eventually results in the stunted growth of the plant or in worse case scenarios can also kill them.

3. MATERIALS AND METHODS

3.1. Significance of Nutrient tablets

The importance of processing nutrients into biochar tablets is to minimize the losses of the excess nutrients which eventually contaminates the atmosphere as well as the freshwater bodies and this can be done by addition of certain binders and fillers.

3.2. Sample preparation and experimental site

Biochar was purchased from the local market. Solid pieces that were fully converted into biochar were separated from the partial ones. Selected pieces were broken down into smaller pieces using a hammer. Afterwards they were passed through a lab scale grinder and converted into powder. The powder of biochar was passed through a sieve (Royal INC, Mesh number 30). The sieved powder was packed into separate plastic bags each weighing 20 grams. The packed powder was stored at room temperature.

Different experimental runs were conducted on different punch pressures (low, medium, and high). Direct compression of biochar powder at different punch pressures was checked. Addition of binding agent (starch solution) at different concentrations and amount was tested. Further addition of filler (Tragacanth gum) at different amounts was also tested.

The experiments were conducted at Atta ur Rehman School of Applied Biosciences(ASAB), National University of Science and Technology (NUST), Islamabad, Pakistan.

3.3. Optimization for formulation of the tablets

The initial optimization experiments included only biochar powder without any binding agent or filler. The tablet machine was set on low pressure. There was no tablet formation on low, medium, or high pressure due to no binding capacity of biochar powder. Then the addition of binder and filler resulted in tablets with low binding capacity (tragacanth gum used as filler and

starch solution was used as binding agent with magnesium stearate as lubricating agent). Various trial experiments were run with different amounts of biochar powder, filler, binding agent and lubricating agent so that the best combination can be obtained which will result in a tablet which is compact, circular in shape, without requiring any dedusting.

Experiments were designed using Random Block Design(RBD) as shown in Table 2.

Table 2: Random Block Design experiments of Biochar tablets.

ID	Biochar (gm)	Filler (gm)	Binding agent (%)	Lubricating agent (gm)	Resting Period (Mins)	Observed Compactness	Dedusting
T ₁	14	6	10	2	45	Very little	Required
T ₂	14	6	10	1	45	Very little	Required
T ₃	13	7	10	2	45	Not compact	Required
T ₄	13	7	10	1	45	Very little	Required
T ₅	14	6	13	2	75	Not satisfactory	Required
T ₆	14	6	13	1	75	Not satisfactory	Required
T ₇	13	7	13	2	75	Relatively compact	Required
T ₈	13	7	13	1	75	Relatively compact	Required
T ₉	14	6	15	2	105	Excellent	Not Required
T ₁₀	14	6	15	1	105	Very good	Very Little
T ₁₁	13	7	15	2	105	Good	Very Little
T ₁₂	13	7	15	1	105	Good	Very Little

3.4. Single punch tablet press machine THDP-3

Single punch tablet machine (Fig 1) is a device that is equipped with two punches (upper and lower punch) that compresses powder in a single die producing uniform tablets of same size, weight and shape. Tablet formation is achieved by passing the powder into a cavity known as die and then the punches are pressed together to fuse the material together. The hopper is a device equipped on the machine that removes the excess powder from top of the die, and to prevent spillage the lower punch moves down into the die. The hopper also delivers the powder into the die cavity and the upper punch is brought down for compression. The compression force makes a strong hard tablet and after compression the lower punch is brought up for ejection of tablet.

3.5. Steps of making Nutrient tablets

After the optimization of the ingredients as shown in the table above, (T₉) shows the best result. Taking this into the account same amounts of the ingredients were used to proceed further for adding nutrients into the biochar tablet.

3.5.1. Calculations of the nutrients to be added individually

As micronutrients selected were Copper (Cu), Boron (B), and (Manganese), were selected, their optimal range in the soil was searched in the research papers. Optimal ranges were checked after that the compound which was to be used as a source for that specific micronutrient was selected and then the further calculations for each mentioned micronutrient were done.

Table 3: Optimal ranges of elements in the soil is shown in the table below.

Serial No	Source of Element	Element	Optimal range of element in soil
1	Boric acid	Boron	10–300mg/Kg (Bauer, 2020)
2	Copper Sulphate	Copper	5-70mg/Kg (Chiou & Hsu, 2019)
3	Manganese Sulphate	Manganese	30-500mg/Kg (Mulder & Gerretsen, 1952)

Calculation for Copper:

Molar Mass of $\text{CuSO}_4 = 249.69\text{g/mol}$

1 tablet should contain 5mg of copper

% Composition of copper in copper (II)Sulphate Pentahydrate = $63.546/249.69 \times 100$

$$= 25.45\%$$

25.45% copper is present in 1 mole of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

I.e.

100g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ gives 25.45g of copper

1 Batch of tablet making gives 25 tablets

25450mg Cu = 100000mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

1mg Cu = $100000/25450$ mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

5 mg Cu = $100000/25450 \times 5$ $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

5mg Cu = 19.65 mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

$$= 0.01965 \text{ g } \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$$

We can conclude that if we use 19.65mg $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, in making of 1 Tablet it will give us presence of 5mg Cu in one tablet.

As we know 1 batch of tablet making gives as 25 tablets approximately, so we multiplied the amount of copper sulfate needed for 1 tablet with 25 and got the value of copper sulfate i.e., 0.5g, which will be added into the mixture so that when we get the batch of 25 tablets, each tablet would contain 5mg of copper approx.

Calculation for Boron:

Molar Mass of $\text{H}_3\text{BO}_3 = 61.83\text{g/mol}$

1 tablet should contain 10mg of boron

% Composition of boron in boric acid = $10.11/61.83 \times 100$

$$= 17.48\%$$

17.48% boron is present in 1 mole of H_3BO_3

I.e.

100g of H_3BO_3 gives 17.48g of boron

1 Batch of tablet making gives 25 tablets

17480mg B = 100000mg H_3BO_3

1mg B = $100000/17480$ mg H_3BO_3

10mg B = $100000/17480 \times 10$ H_3BO_3

10mg B = 57.2 mg H_3BO_3

$$= 0.0572 \text{ g } \text{H}_3\text{BO}_3$$

We can conclude that if we use 57.2mg H_3BO_3 , in making of 1 Tablet it will give us presence of 10mg B in one tablet.

As we know 1 batch of tablet making gives as 25 tablets approximately, so we multiplied the amount of boric acid needed for 1 tablet with 25 and got the value of boric acid i.e., 1.4g, which will be added into the mixture so that when we get the batch of 25 tablets, each tablet would contain 10mg of boron approx.

Calculation for Manganese:

Molar Mass of $\text{MnSO}_4 = 169.02\text{g/mol}$

1 tablet should contain 30mg of manganese

% Composition of manganese in manganese sulfate = $54.94/169.02 \times 100$

$$= 32.5\%$$

32.5% manganese is present in 1 mole of MnSO_4

I.e.

100g of MnSO_4 gives 32.5g of manganese

1 Batch of tablet making gives 25 tablets

32500mg Mn = 100000mg MnSO_4

1mg Mn = $100000/32500$ mg MnSO_4

30mg Mn = $100000/32500 \times 30$ MnSO_4

30mg Mn = 92.3 mg MnSO_4

$$= 0.0923 \text{ g } \text{MnSO}_4$$

We can conclude that if we use 92.3mg MnSO_4 , in making of 1 Tablet it will give us presence of 30mg Mn in one tablet.

As we know 1 batch of tablet making gives as 25 tablets approximately, so we multiplied the amount of manganese sulfate needed for 1 tablet with 25 and got the value of manganese sulfate i.e., 2.3g, which will be added into the mixture so that when we get the batch of 25 tablets, each tablet would contain 30mg of manganese approx.

3.5.2. Biochar grinding

Biochar was bought and was manually broken down into small pieces with a hammer. Afterwards, small pieces of Biochar were passed through a lab scale grinder.

3.5.3. Adjustment of punch pressure

The pressure of single punch THDP-3 tableting machine was adjusted manually to the ideal pressure point. The bolt of the upper punch was rotated clockwise to increase the pressure.

3.5.4. Filler

Tablets breakdown immediately upon pressure release irrespective of the pressure and amount of binder. This happens because the binders just fill the holes rather than forming bridges that connect the biochar particles. Filler forms the bridges that connect the binder and biochar particles. Organic compound tragacanth gum was used as a filler. The filler was mixed with biochar powder in dry mixing.

3.5.5. Addition of nutrients

After mixing of the biochar powder with the filler using the composition from selected from the (T₉) experiment, copper sulfate, boric acid and manganese sulfate in salts form was added as a source for copper, boron, and manganese respectively, to the mixture of biochar and filler. This was the step of dry mixing. It is important to be noted that different batches were made of nutrient tablets where the composition from the (T₉) experiment was kept constant and in 1st batch copper sulfate was added which gave us copper nutrient tablets, in 2nd batch boric acid was added which gave us boron nutrient tablets and in 3rd batch manganese sulfate was added which gave us manganese nutrient tablets.

3.5.6. Binding agent

Binding agent has the most important role in tablet formation as they bind the ingredients of the tablet together. The binding agent also help to convert powder into granules. Organic binding agents such as starch solution was added to the mixture of filler and biochar powder and salt.

3.5.7. Granulation

The granulation of powder mixture consisting of filler, salt and binding agent added to biochar powder is passed through a sieve and small granules are created. This step ensures the proper flow of mixture through the press machine as well as compressibility of tablets so that the tablets formed remain compact and stable after being compressed.

3.5.8. Lubricating agent

The last step before passing the mixture through a compression machine is the lubrication of the mixture to ensure nonstop flow of the powder. Lubricating agents such as magnesium stearate was added.

3.6. Hardness Test

The hardness of the tablets was calculated using the Universal Testing machine (AGX-PLUS model, Shimadzu 50Kn cell, Japan) (Fig 2). The tablets were placed under the press, and it was run at a speed of 1.0 mm per minute. Once the tablet deformed the machine was stopped automatically and the reading was noted.

3.7. Estimation of Dissolution time

Dissolution is a process of dissociating /disintegrating/dispersing of a solute into a solvent, resulting in the formation of a molecular level, physically and chemically homogenous dispersion. Unlike solubility, it is a process.(Byrn et al., 2017).

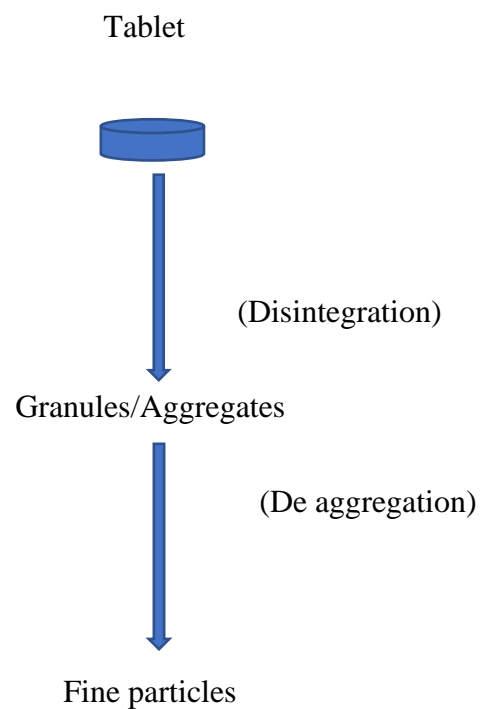


Figure 1: Summarizing the process of dissolution where it is shown in detail how when a tablet is placed in a liquid it first dissociates into smaller particles and eventually, they are broken down into further finer particles, hence completing the process of dissolution.

3.8. FTIR analysis

Fourier transform infrared spectroscopy (FTIR), is a technique which is used for the identification and confirmation of the compounds present in a mixture. The basic working principle of this technique is identifying functional groups which are present within the molecules. Vibration of these functional groups takes place when they are irradiated with specific wavelengths of light. This vibration of the functional groups takes place either through bending or stretching. These vibrations of the functional groups and their intensity (also known as % transmission), are plotted on the graph against light's frequency (cm^{-1}), to which sample has been exposed, eventually producing a FTIR spectrum. Some portions of the produced FTIR spectrum are unique and are known as fingerprint region. Major advantage of FTIR spectroscopy is that it does not require derivatization and is non-destructive. (Daéid, 2004).

3.9. XRD analysis

X-Ray Diffraction (XRD) is used to analyze polycrystalline or single crystal materials. Major use of XRD is characterization and identification of the compounds based on their diffraction pattern. Working basis of the XRDs is on reflection geometry, where source of the X-ray along with the detector is placed on the same side where sample is present. Scattered X-rays from the source reflects through the sample and are detected onto the detector. Constructive interference takes place between the scattered X-rays. The interference is based upon the Bragg-Brentano law for determining the characteristics of the sample for which XRD analysis is carried out. XRD powder diffractometer consists of a source, a detector, sample part, primary optics, and secondary optics. The output we get after the XRD analysis is known as a diffractogram, in which x -axis shows the functioning of the scanning angle while y -axis represents the intensity. XRD is used for characterizing the physical structures (crystalline/or amorphous) and the composition of the given samples. (Gumustas et al., 2017)

4. RESULTS

4.1. Interpretation of Hardness test

As with the experiments run from (T₁ - T₁₂), it was noted that experiments from (T₉) shows the best results, so carrying forwards those amounts of the components for the nutrient tablets we moved further making the nutrient tablets of copper, boron, and manganese. The composition taken from the (T₉) experiment were 14g biochar, 6g tragacanth gum (filler), 10ml of the 15% starch solution i.e., the binding agent and 2g of lubricating agent i.e., magnesium stearate. While making these tablets, in the step of granulation, resting period provided was of 1hr and 45 minutes, and as a result, tablets were formulated which were excellent in shape, were not broken into pieces when fell into the container from the machine and most importantly no dedusting was required. With this composition, the formulated tablets of copper, boron, and manganese, 4 tablets from each of the tablets' batch were chosen for the hardness test and the mean value of their hardness was noted as shown in the table no 4.

Plant-Nutrient Tablets	Quantity of Samples	Mean Hardness (N/mm²)
Copper-nutrient tablet	Cu ₁	2.2
	Cu ₂	
	Cu ₃	
	Cu ₄	
Boron-nutrient tablet	B ₁	2.1
	B ₂	
	B ₃	
	B ₄	
Manganese-nutrient tablet	Mn ₁	2.3
	Mn ₂	
	Mn ₃	
	Mn ₄	

Table 4: Mean hardness values of the copper-nutrient tablets, boron-nutrient tablets, and manganese-nutrient tablets is shown in tabular form.

It is also assumed that when the amount of tragacanth gum in decreased quantity is used with 15% starch solution observed hardness of the tablets was also increased as well as their shape and no need of dedusting was required as also mentioned before.

It was noted that the hardness was indirectly proportional to amount of filler in case of tragacanth gum. decreasing the amount of tragacanth gum, the hardness of the tablets increased. Tragacanth gum proved to be a good filler for hardness in low quantity with the 15% binding agent i.e., starch solution. Compactness and hardness of the tablets was one of the very important factors in our study. The hardness test was conducted keeping in view the prospects of the study and the obtained results also shows that tablets formulated were compact in shape and hard enough that after proper packaging, they can easily be transported from one place to another even to longer distances without the tablets being cracked or broken.

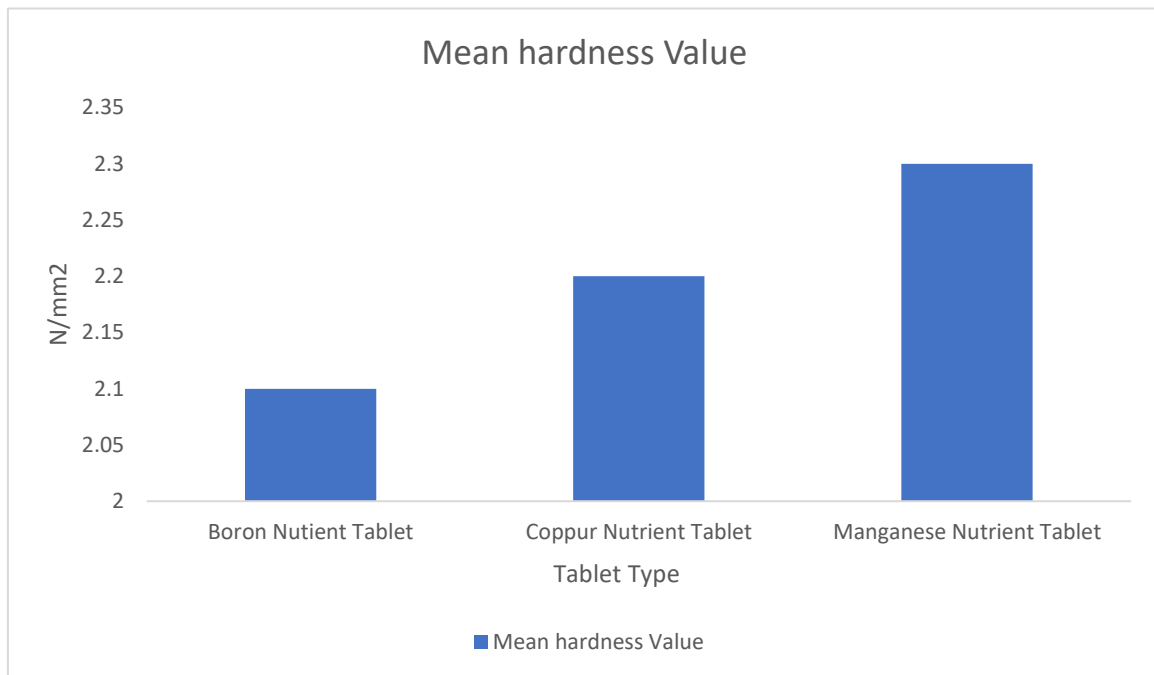


Figure 2: Mean hardness values of Boron-nutrient tablets, copper-nutrient tablets, and manganese-nutrient tablets are shown in the figure above in form of bar charts.

4.2. Qualitative Interpretation of Dissolution analysis

Considering the concept of dissolution as discussed above in Chapter 3 Materials and Methods, qualitative dissolution analysis of the tablets was conducted keeping in view the prospects of the nutrient tablets. Disintegration mechanism of the tablet means breaking up of tablet which is in compressed form into smaller granules i.e., breakage of the inter-particulate bonds that were formed during the process of formulating the tablets. There is a need of a force during the process of disintegration for the disruption of the bonds. The most important step in the process of disintegration is the penetration of the liquid into the compact mixture. It is also important to be clear that it's not the penetration power of the liquid which helps in building up of the pressure for rupturing the particle-particle bonds, instead liquid penetration is the preliminary step for initiating other mechanisms involved in disintegration process like swelling. Mechanism of swelling is involved in the process of disintegration. (Markl & Zeitler, 2017). Swelling means enlargement of the particles of the tablet is omni-directional, which results in the building up of the pressure which push apart the particles which are adjoined, leading to stresses which are exerted and eventually resulting in the breakage of the tablet.

Keeping in view the prospects of the study, this qualitative study of the dissolution is very important as we can conclude that this tablet takes more than 3 months' time for disintegrating into the fine particles in a flask of tap water filled with 500ml. For this, with (T₉) experiment's composition, the formulated tablets of copper, boron, and manganese, 4 tablets from each of the tablets' batch shown in figure 5, were chosen for the qualitative dissolution analysis as shown in the table 5 and also in the form of bar charts for better understanding in figure 6.



Figure 3: Copper-nutrient tablets, boron-nutrient tablets, and manganese-nutrient tablets placed in 500ml flasks filled with tap water are shown.

Table 5: Mean days taken by the copper-nutrient tablets, boron-nutrient tablets, and manganese-nutrient tablets for the process of dissolution is shown in tabular form.

Nutrient Tablet	Quantity of Samples	Mean time taken for disintegration (Days)
Copper-nutrient tablet	Cu ₁	100
	Cu ₂	
	Cu ₃	
	Cu ₄	
Boron-nutrient tablet	B ₁	102
	B ₂	
	B ₃	
	B ₄	
Manganese-nutrient tablet	Mn ₁	101
	Mn ₂	
	Mn ₃	
	Mn ₄	

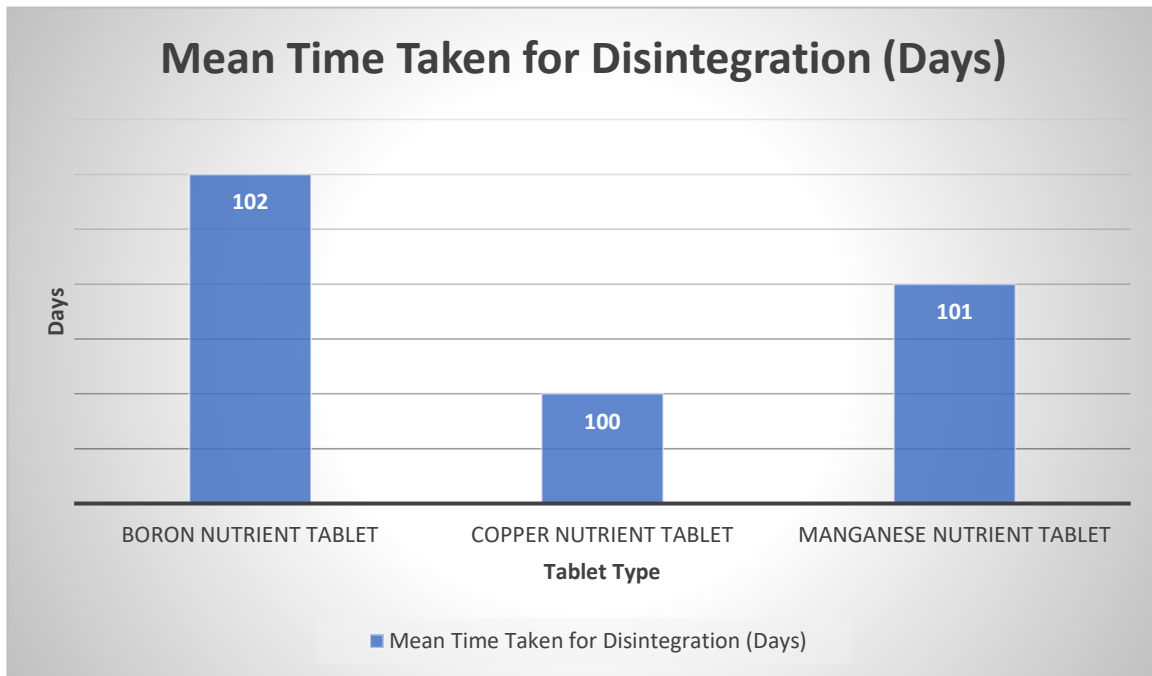


Figure 4: Mean time taken by copper-nutrient tablet, boron-nutrient tablet, and manganese-nutrient tablet for the process of dissolution is shown in form of bar charts.

4.3. Interpretation of FTIR analysis

The basic working principle of this technique is identifying functional groups which are present within the molecules. In the FT-IR spectrum of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, characteristic peaks are obtained at 3114, 1667, 1063 and 860 cm^{-1} . Peaks which are obtained over 3000 cm^{-1} explain the crystal water's presence in the structure, while the band values obtained at lower end explain the occurrence of the vibrations between nonmetal atoms and O. (Derun et al., 2014).

In the FT-IR analysis of boric acid characteristic peaks are obtained at 3250 and 2970 cm^{-1} referring to stretching of (OH) and (CH) groups respectively while stretching vibration at the band value of 1480 cm^{-1} refers to that of (BO) functional group. (Mondal & Banthia, 2005) (Fang et al., 2016).

According to the FT-IR analysis of manganese sulfate performed by the National Institute of Standards and Technology (NIST), characteristic peaks were obtained at 2990 cm^{-1} , 1490 cm^{-1} , 1210 cm^{-1} , 1000 cm^{-1} , 846 cm^{-1} , and 610 cm^{-1} .

In the FT-IR spectrum of magnesium stearate, twin peaks which are obtained at 1466 and 1577 cm^{-1} are attributed to stretching vibration of symmetric carboxylate (COO^-), and stretching vibration of asymmetric carboxylate, respectively. Broad band obtained at 3452 cm^{-1} refers to stretching vibrations due to (OH) whilst obtained peaks at 2850 and 2917 cm^{-1} refers to stretching vibration of (CH). (Nep & Conway, 2012).

In the FT-IR spectrum of starch broad band obtained at 3393 cm^{-1} refers to (OH) groups stretching mode. Adsorption band peak at 1648 refers to the intermolecular *H-Bond* which involves the carboxyl group, whilst band peaks at 2931 and 1155 cm^{-1} are referred to as (CH) and (CO) stretching respectively. (Hebeish et al., 2009).

In the FT-IR analysis of biochar peak at 3400 cm^{-1} represents the stretching vibrating of (OH) group. Peak at 1628 cm^{-1} represents stretching vibration of asymmetric C-O group.(Wang et al., 2020). Carbonyl and carboxyl groups which are present in the biochar are acting as the chelating agents, serving as the enhancers for the ion exchange capacity of biomaterial.(Adeniyi et al., 2021).

FT-IR analysis conducted for the detection of the functional groups in the nutrient tablet was conducted for identifying, detecting, and confirming the presence of the nutrients which were added in the form of salts which includes copper sulfate pentahydrate acting as a source for copper nutrient, boric acid for boron and manganese sulfate monohydrate for manganese.

It is important to note that the nutrient tablet formulated consists of multiple ingredients namely, biochar, tragacanth gum, salt as a source for the nutrient, binding agent, and lubricating agent, so it is important to know that many peaks will be overlapping such as for the stretching vibrations of (OH) and (CH) groups. The reason for this test was to detect the presence of the nutrient in the mixture so that we can be confident enough that the nutrient tablet we formulated contains the desired nutrient and then we can move further with the field/pot experiments, keeping in view the prospects of the study.

Cu tablet:

The FT-IR of the copper nutrient tablet was conducted, and characteristic peaks of the functional groups were obtained at the band value 3419 cm^{-1} , 2922 cm^{-1} , 1619 cm^{-1} , 1447 cm^{-1} , 1116 cm^{-1} and 864 cm^{-1} as shown in the figure below were attributed to the stretching vibrations of *OH*, *CH*, *COO*⁻, *COO*⁻, *CO* and *Cu-S* respectively.

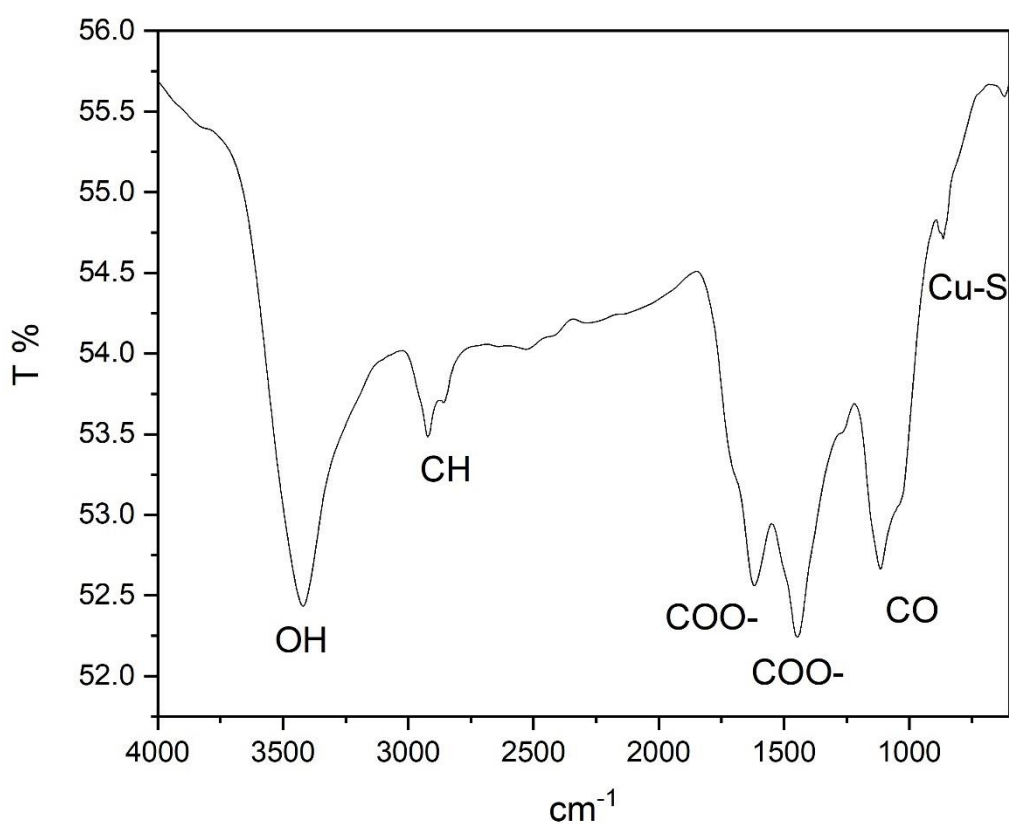


Figure 5: FT-IR spectrum for copper-nutrient tablet above shows characteristic peaks of the functional groups were obtained at the band value 3419 cm^{-1} , 2922 cm^{-1} , 1619 cm^{-1} , 1447 cm^{-1} , 1116 cm^{-1} and 864 cm^{-1} .

B tablet:

The FT-IR of the boron nutrient tablet was conducted, and characteristic peaks of the functional groups were obtained at the band value 3426 cm^{-1} , 2919 cm^{-1} , 1624 cm^{-1} , 1456 cm^{-1} and 1050 cm^{-1} as shown in the figure below were attributed to the stretching vibrations of *OH*, *CH*, *COO*⁻, *B-O* and *CO* respectively.

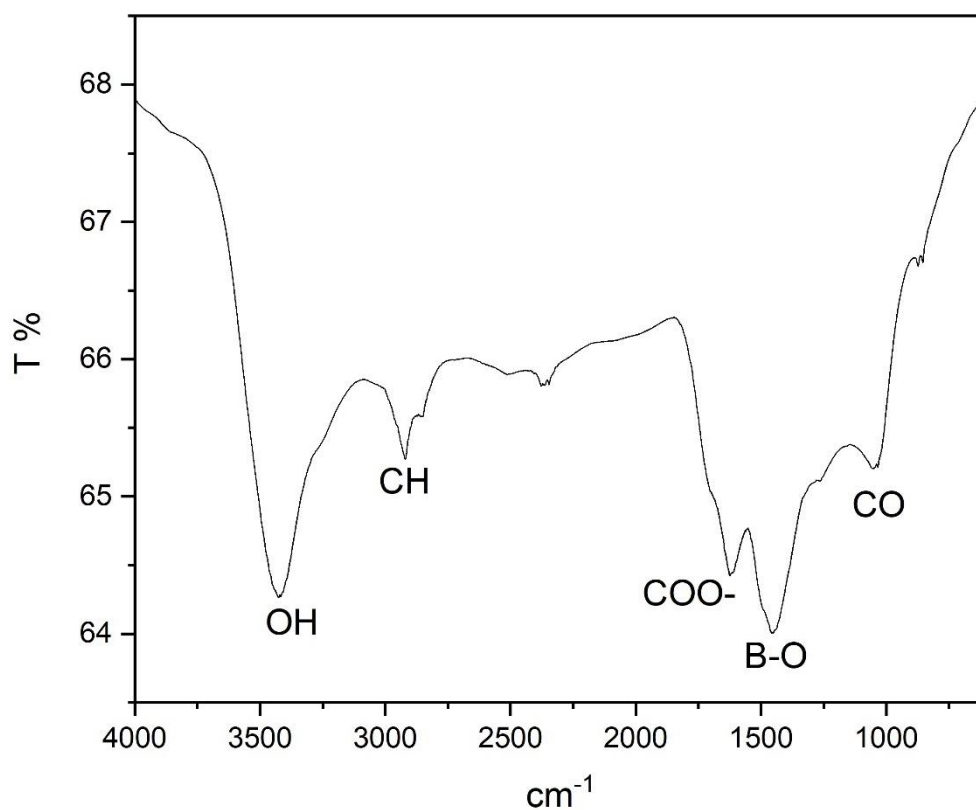


Figure 6: FT-IR spectrum for Boron-nutrient tablet above shows characteristic peaks of the functional groups were obtained at the band value 3426 cm^{-1} , 2919 cm^{-1} , 1624 cm^{-1} , 1456 cm^{-1} and 1050 cm^{-1} .

Mn tablet:

The FT-IR of the manganese nutrient tablet was conducted, and characteristic peaks of the functional groups were obtained at the band value 3423 cm^{-1} , 2927 cm^{-1} , 2382 cm^{-1} , 1629 cm^{-1} , 1447 cm^{-1} and 1114 cm^{-1} as shown in the figure below were attributed to the stretching vibrations of *OH*, *CH*, *COO*⁻, *COO*⁻, *CO* and *Mn-S* respectively.

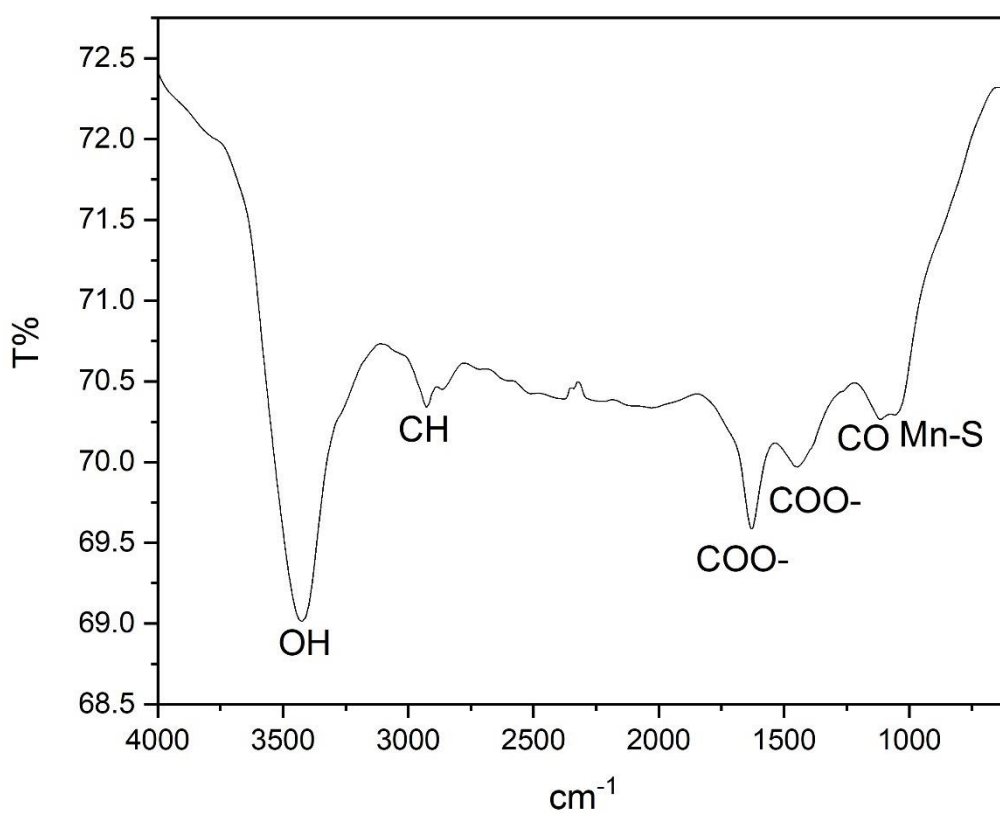


Figure 7: FT-IR spectrum for Manganese-nutrient tablet above shows characteristic peaks of the functional groups were obtained at the band value 3423 cm^{-1} , 2927 cm^{-1} , 2382 cm^{-1} , 1629 cm^{-1} , 1447 cm^{-1} and 1114 cm^{-1} .

4.4. Interpretation of XRD analysis

Use of XRD is characterization and identification of the compounds based on their diffraction pattern. Working basis of the XRDs is on reflection geometry, where source of the X-ray along with the detector is placed on the same side where sample is present. Scattered X-rays from the source reflects through the sample and are detected onto the detector. Constructive interference takes place between the scattered X-rays. The interference is based upon the Bragg-Brentano law for determining the characteristics of the sample for which XRD analysis is carried out. XRD powder diffractometer consists of a source, a detector, sample part, primary optics, and secondary optics. The output we get after the XRD analysis is known as a diffractogram, in which x -axis shows the functioning of the scanning angle while y -axis represents the intensity. XRD pattern of copper sulfate pentahydrate shows total of three major peaks at the $^{\circ}2\theta$ values of 48.5, 16.125 and 18.8.(Derun et al., 2014). XRD pattern of boric acid shows peaks at the $^{\circ}2\theta$ values of 28°, 13°, 31°, 32°, 34°, 44°.(Huber et al., 2019)(Bingöl & Çopur, 2019)(Atala et al., 2020)(Sheikh et al., 2017)(Bu et al., 2018). XRD pattern of manganese shows peaks at the $^{\circ}2\theta$ values of 15°, 22°, 32°, 33°, 35°, 43°, 49°.(Lin et al., 2016)(Y. H. Lee et al., 2020). XRD pattern of starch shows peaks at the $^{\circ}2\theta$ values of 15°, 18°, and 23°.(de Vasconcelos et al., 2015)(Zuo et al., 2014). XRD pattern of biochar shows peaks at the $^{\circ}2\theta$ values in a range from 20°-30°.(Y. Liu et al., 2012). XRD pattern of magnesium stearate shows peaks at the $^{\circ}2\theta$ values of 6° and 10°.(Focke et al., 2014). XRD analysis was conducted for the detection of the materials present in the nutrient tablet. As it is discussed above that the nutrient tablet formulated consists of multiple ingredients namely, biochar, tragacanth gum, salt as a source for the nutrient, binding agent, and lubricating agent, so it is important to know that many peaks will be overlapping or may be very close to each other which may sometimes prove to be a difficult task for identifying the peaks individually. The most important reason for this test was to detect the presence of the nutrient in the mixture so

that we can be confident enough that the nutrient tablet we formulated contains the desired nutrient and then we can move further with the field/pot experiments, keeping in view the prospects of the study.

Cu tablet:

The XRD analysis of copper tablet shows characteristics peaks at the $^{\circ}2\theta$ values of 6° and 10° representing the presence of magnesium stearate, at 15° and 23° representing starch, at 26° and 29° representing biochar, and at 16° , 18° , and 48° representing copper sulfate pentahydrate as shown in the figure 10.

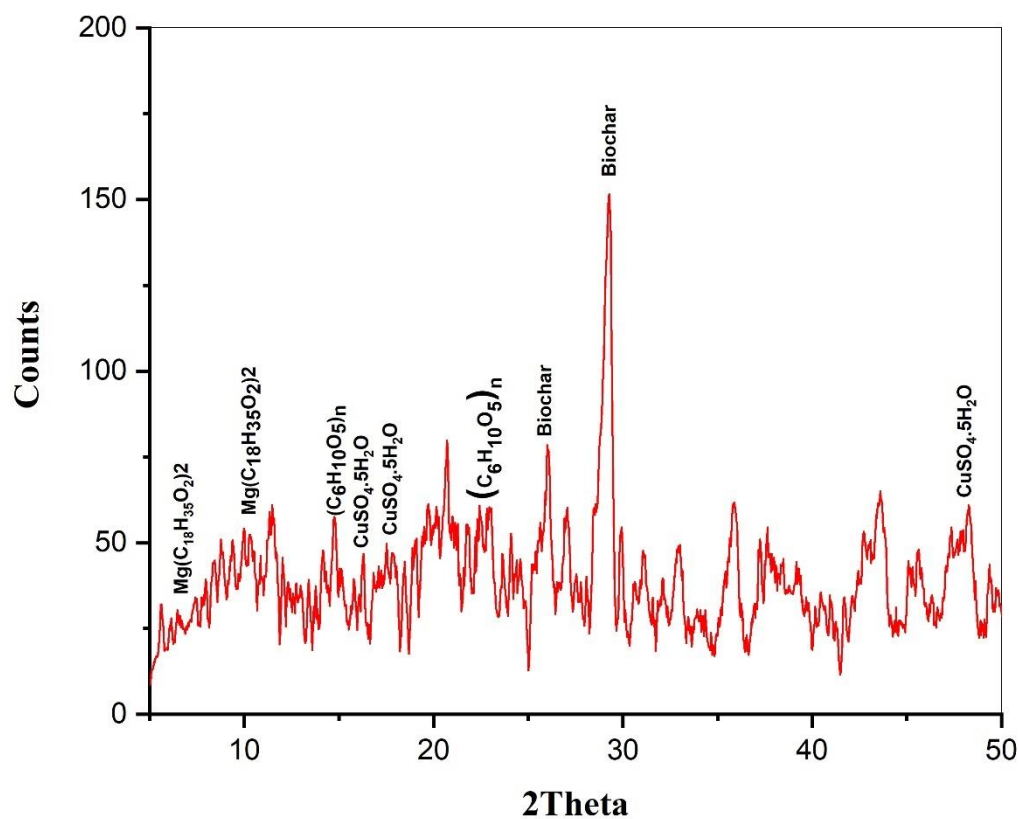


Figure 8: XRD peaks for copper-nutrient tablet above shows characteristics peaks at the $^{\circ}2\text{Theta}$ values of 6° and 10° representing the presence of magnesium stearate, at 15° and 23° representing starch, at 26° and 29° representing biochar, and at 16° , 18° , and 48° representing copper sulfate pentahydrate.

B tablet:

The XRD analysis of boron tablet shows characteristics peaks at the 2θ values of 6° and 10° representing the presence of magnesium stearate, at 15° and 23° representing starch, at 20° and 26° representing biochar, and at 14° , 28° , 32° , 36° , 38° and 44° representing boric acid as shown in the figure 11.

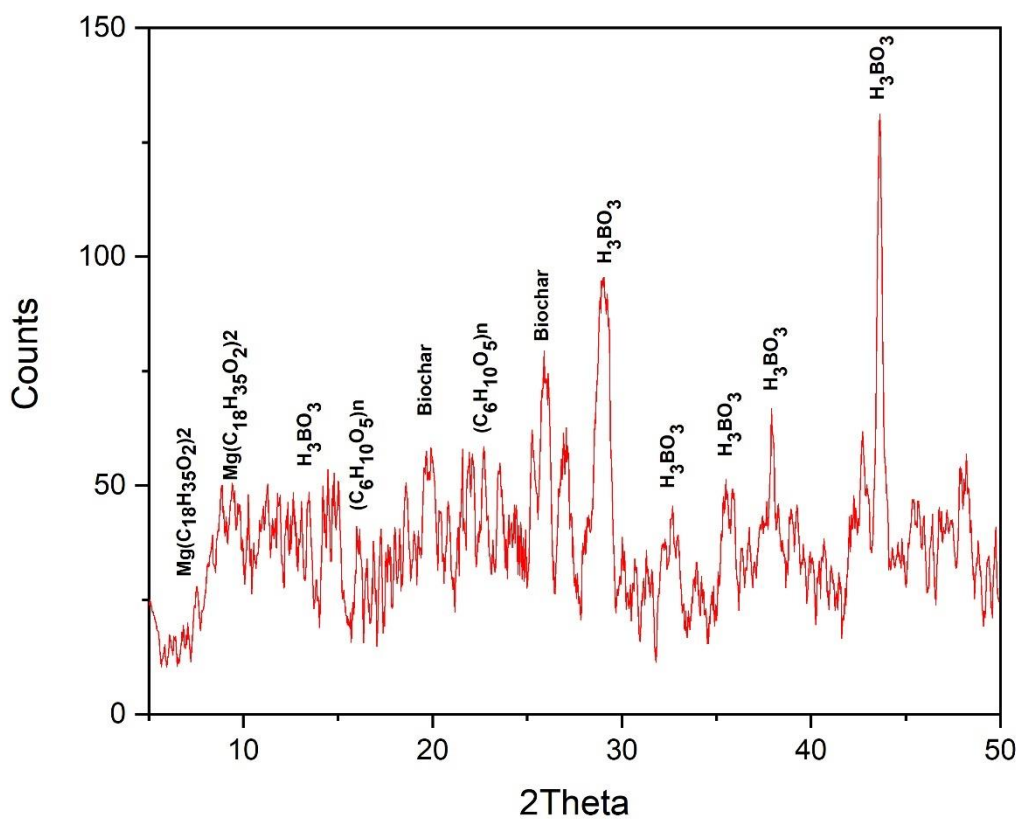


Figure 9: XRD peaks for Boron-nutrient tablet above shows characteristics peaks at the 2θ values of 6° and 10° representing the presence of magnesium stearate, at 15° and 23° representing starch, at 20° and 26° representing biochar, and at 14° , 28° , 32° , 36° , 38° and 44° representing boric acid.

Mn tablet:

The XRD analysis of manganese tablet shows characteristics peaks at the $^{\circ}2\theta$ values of 6° and 10° representing the presence of magnesium stearate, at 15° and 23° representing starch, at 26° and 29° representing biochar, and at 12° , 21° , 31° , 33° , 36° , 43° , and 48° representing manganese sulfate monohydrate as shown in the figure 12.

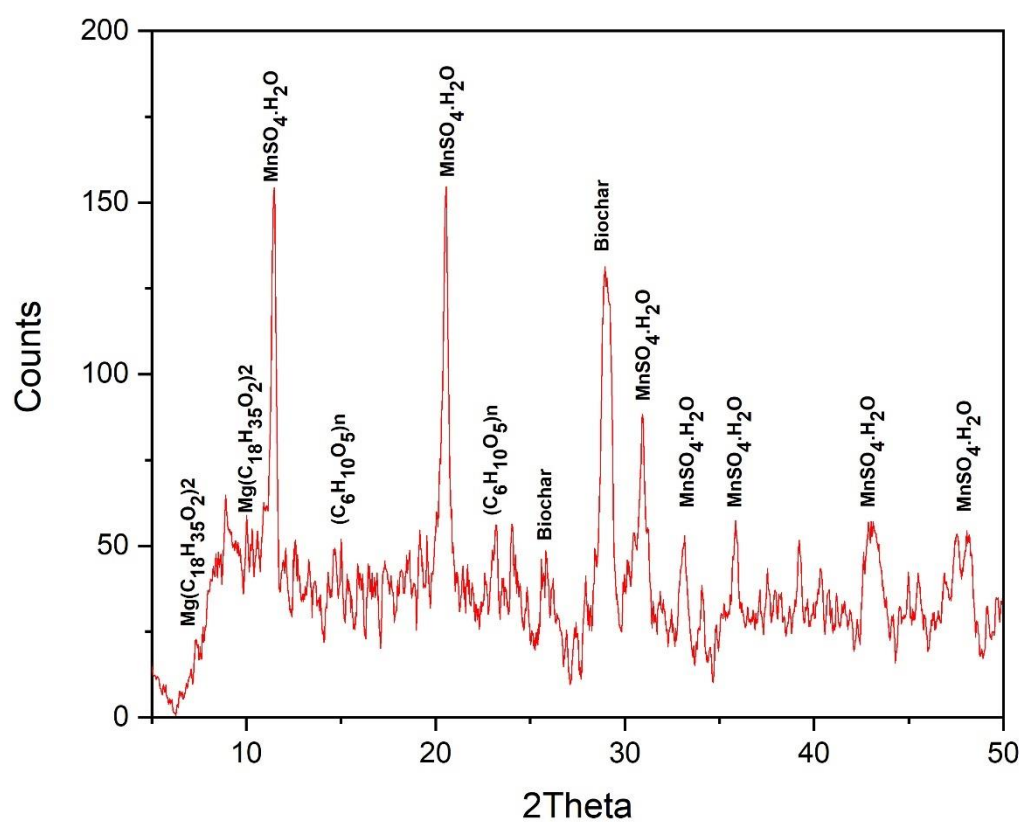


Figure 10: XRD peaks for manganese-nutrient tablet above shows characteristics peaks at the $^{\circ}2\theta$ values of 6° and 10° representing the presence of magnesium stearate, at 15° and 23° representing starch, at 26° and 29° representing biochar, and at 12° , 21° , 31° , 33° , 36° , 43° , and 48° representing manganese sulfate monohydrate

5. DISCUSSION

Population of the world is expected to touch 9.5 billion by year 2050.(Kareem et al., 2021). Degradation caused by heavy flooding, desertification and industrialization have resulted in the diminishing of the arable land and it is seen as a big threat to world's food security.(Jie et al., 2002). Due to all these issues food requirements are also expected to double globally by 2050.(Brown et al., 2009). For resolving such issues and to improve and modify our agricultural sector steps are taken which can help in making agricultural sector more sustainable and successful. Despite using conventional fertilizers in larger quantities, low utilization of nutrients by the crop is observed with subsequent loss into environment through denitrification, leaching and surface runoff.(Azeem et al., 2016). Directly administrating plants with fertilizers shows that the plants' efficiency of utilizing the fertilizers is only 30-35%.(Lawrencia et al., 2021). Due to loss of fertilizer in this manner nutrient availability to plants is diminished or deprived in some cases, process cost increases and environmental damage is also done.(Li et al., 2018). To overcome these challenges slow-release fertilizers (SRFs) and controlled-release fertilizers (CRFs), are being produced and used.(Trenkel, 2013). Studies have revealed that SRFs and CRFs can contribute into the efficiency of nutrient use and reducing the environmental damage as it is eco-friendly with minimal pollution.(Roshanravan et al., 2015)(Tian et al., 2021). The idea is to provide the plants with the nutrient tablet that can help the plant in overcoming the deficiencies caused due to insufficient amount of micro and macro-nutrients. Fertilizers in tableted form has been used in past. Formulation of densified biochar loaded with separate nutrients into the form of tablets proves to enhance the conveyance and handling efficiency of formulated biochar nutrient tablets or can also be called plant-nutrient tablets.(Y. L. Lee et al., 2021). Carrying forward the idea presented in this current work, basing upon the extensive literature review the biochar loaded plant-nutrient tablets for copper, boron, and manganese were formulated.

The mean hardness values and mean time taken by the plant-nutrient tablets for the process of disintegration is shown above in the Table 3 and 4 respectively. The hardness of the nutrient tablets was characterized as having good mechanical strength as the maximum strength was around 3.8N/mm^2 . The mean values obtained from the hardness test were very satisfying as they fulfilled the reason behind conducting the hardness test which was transportation and to confirm that the tablets are in good shape i.e., they look aesthetically good as well as hard enough that they can be transported through far areas after proper packaging without resulting in any breakage of the tablets. Binding agents play an important role in the formation of tablets as it provides the essential cohesiveness between the solid particles and bonds them together as a solid tablet when compressed. The relation between the particles of biochar and binding agent is purely physical. The properties of the binding agent do not change at the surface of the biochar particles due to solubility. Starch solution was used as the binding agent which has good binding relations with the biochar powder. The particles of starch have suitable viscosity when used in wet granulation and the particles increase the hardness after they have been dried. Starch paste in concentrations from 5%-20% can be used in formulation of the tablets. (Manek et al., 2012). The binding agent concentration has also been directly involved in the dissolution analysis i.e., if binding agent is used in low concentrations, then the tablets will not be hard enough and upon putting it in the flask filled with 500ml tap water will disintegrate very quickly, failing the objective of slow release of the nutrient. When the tablets were formulated using the compositions from (T₉) experiment hard and aesthetically good tablets were formulated which stayed in the 500ml tap water for longer time as shown above in the table 4. Once now we have the average time for the nutrient tablets i.e., for how long they can stay in the tap water, we can design the experiment to check the release of the nutrient tablets which is the prospect of this study.

FT-IR and XRD analysis summed up the presence of the salts which were added as the source of the nutrients during the formulation of the tablets. These tests were conducted for confirming the presence because there were chances that maybe during the tablet formulation the salt powders may get lost in the machine or for may be many other numerous reasons as the salt powders are added in very minute quantities as shown above in section 3.5.1.

To sum it up we can conclude that the nutrient tablets formulated are hard enough fulfilling the objective of transportation, strong enough that they can stay for longer time when applied and the desired nutrients i.e., boron, copper and manganese are not lost during the tablet formulating process and are present in boron-nutrient tablet, copper-nutrient tablet and manganese-nutrient tablet respectively.

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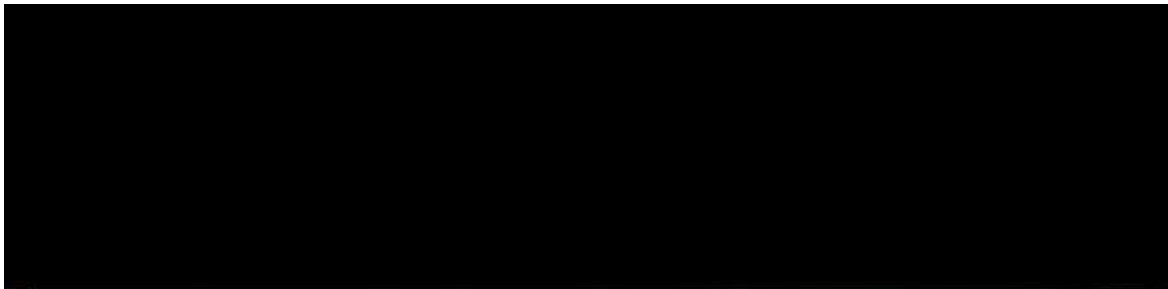
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7. APPENDICES

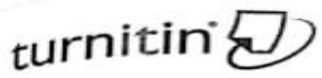
Appendix 1.

Preparation of 15% starch solution

15 grams of starch was weighted and put in a beaker. The volume of beaker was filled with distilled water to the mark of 100 ml. The mixture was continuously stirred until all the starch was dissolved in the water.



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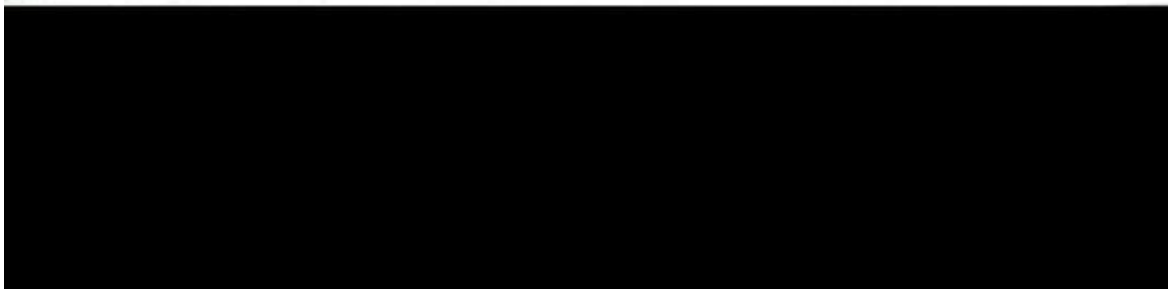


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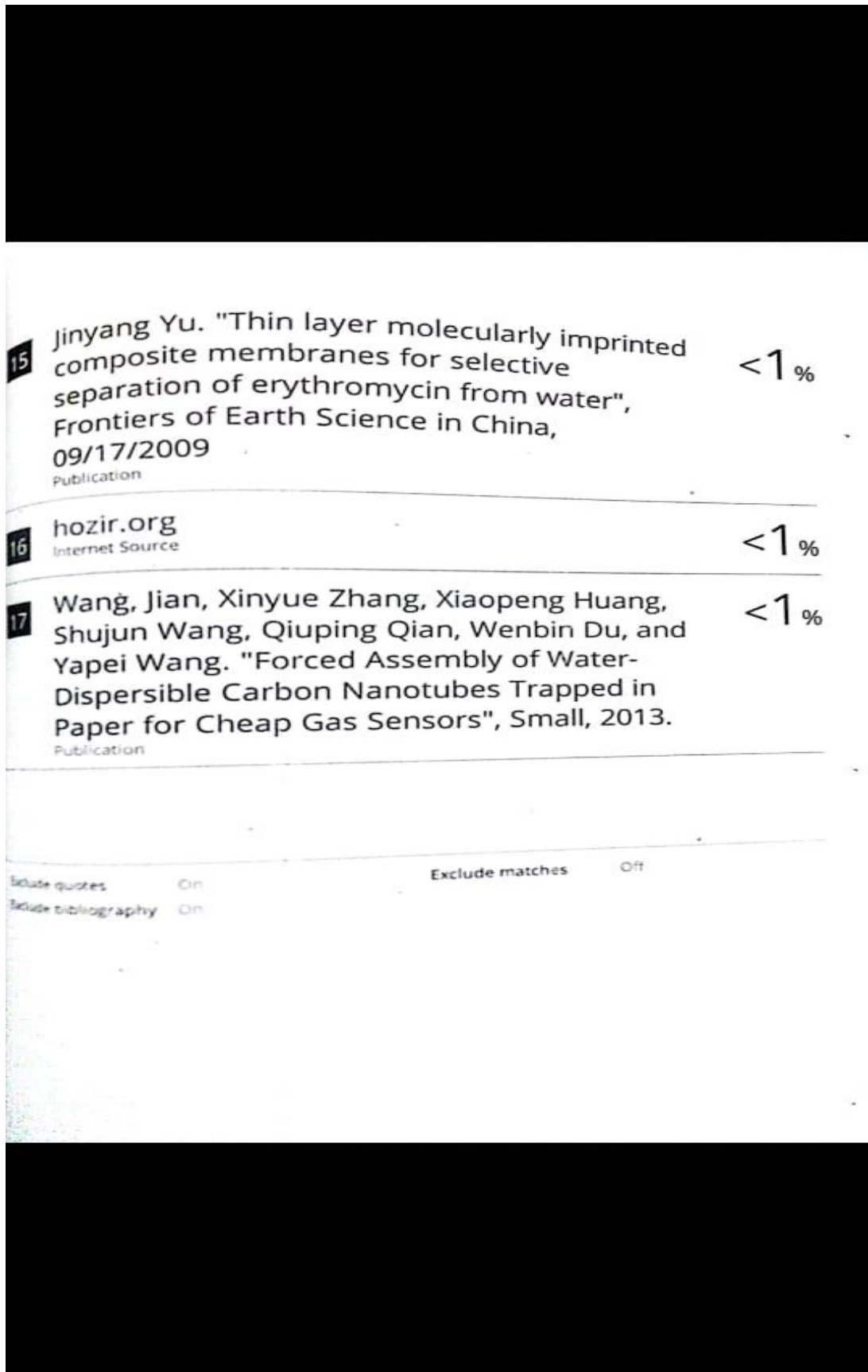
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