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Extraction and Characterization of Okro (Abelmoschus Esculentus) To Be Used As an EOR Fluid

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ABSTRACT-This research deals with extraction and characterization of Abelmoschusesculentussample to be used in Enhanced Oil Recovery (EOR) applications.. Using water based extraction method; characterization of the extracted mucilage was done by various parameters such as micromeritic studies, flow behaviour, organoleptic properties, surface tension, and viscosity, loss on drying, ash value and swelling index. Fourier transform infra-red (FT-IR),Scanning electron microscopy (SEM), and X-Ray Diffraction (XRD) analyses were carried out on Okropowdered sample The FT-IR analysis revealed that the okra powder is composed of organic substances such as polyphenol and carboxyl groups, The okromucilage used in this research were interpreted on the basis of performed XRD analyses and petrographic observations. XRD analysis was carried out using X-ray diffractometryto analyse the phase composition and identify the amorphous nature of the Abelmoschusesculentusmucilage.The diffraction data were collected from 2 to 70^{O} of 2θ starting from 0.02 at 1 second. The morphologies of okro powderedsample was analyzed by SEM. It revealed that theokrosamplewas mostly amorphous in nature.

Keywords (EOR, Okromucilage,FT-IR,XRD,SEM)

I. INTRODUCTION

Natural polymers are generally obtained from plant. They are high molecular weight; water soluble polymers made up of monosaccharide unit and joined by glucosidic bond, (Uzmaet al.,2013), and (Krishna et al.,2011).

Okra (Abelmoschusesculentus L.) is one of the most widely known and utilized species of the family Malvaceae. Economically, okra is an important vegetable crop grown in tropical and sub-tropical regions of the world. Mostly, it is grown for its green leaves and pods as green vegetable according to Naveedet al. (2009). In studies reported by Gopalanet al. (2007), one hundred grams (100 g) of okra contain moisture (89.6 g), minerals (0.7 g), protein (1.9 g/100 g), carbohydrates (6.4 g), fat (0.2 g), calcium (66 mg), fiber (1.2 g), calories (35 mg), potassium (103 mg), phosphorus (56 mg), magnesium (53.0 mg), and sodium (6.9 mg).

Okra gum from the pods of Hibiscus esculentus is one of the advantageous polysaccharides that is currently being studied as a hydrophilic polymer, Zaharuddinet al.(2014). Okra plant grows very fast, is grown in all soil types, and is among the most heat and drought-tolerant vegetables (Bakre and Jaiyeoba, 2009). It has been investigated as a binding agent for tablets and has also been shown to produce tablets with good hardness, friability, and drug release profiles (Okoyeet al.2011). It has advantage over most commercial

synthetic polymers as it is safe, chemically inert, nonirritant, biodegradable, biocompatible, and eco-friendly. Since it is widely harvested and does not require toxicology studies, it is therefore considered to be economical Malviyaet al.(2011). Okra gum contains random coil polysaccharides consisting of galactose, rhamnose, and galacturonic acid. The repeating units of the gum were found to be (1-2)-rhamnose and (1-4)-galacturonic acid residues with disaccharide side chains and a degree of acetylation (DA = 58). When extracted in water, these polysaccharides can produce highly viscous solution with a slimy appearance. Therefore, the highly viscous property of Okra gum may be useful as a retarding polymer.

The well-known natural polymers are aloe mucilage, guar gum, karaya gum, bhara gum,

International Journal of Advances in Engineering and Management (IJAEM)
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sodium alginate, locust bean gum, okra gum and linseed mucilage (Pawanet al., 2001; Sujithaet al., 2012). Gum as obtained from hydrocolloids of plant and can be classified into two groups' i.e.anionic and non-ionic polysaccharides.Hence by modification gum can alter their physicochemical properties (Ogajiet al.,2011). Mucilage is a metabolized product which is intracellularly formed without injury to the plant (Sujithaet al., 2012). Gums are readily soluble in water while mucilage forms slimy mass in the presence of water. Gum and mucilage are translucent, amorphous substances which are produced by plants. They can be used as a thickener,emulsifier, sweetener, viscosity enhancer. The natural polymers find applications in the households, agriculture, food industries and in packaging and they help in decreasing the environmental pollution and resulting in disposal in landfills (Langer and Peppas, 1981). Natural polymers are used as an environment cleaner, renewable and also help in recycling of global carbon (Langer and Peppas, 1981; Malviya, 2010). Okro fruits are rich in vitamins, calcium, potassium, and other mineral matters. The mature okra seed is a good source of oil and protein, and has been known to have superior nutritional quality. Okro seed oil is rich in unsaturated fatty acids such as linoleic acid which is essential for human nutrition. They are also known as ladies finger. It is used as a binder and produces tablet formulations with good and optimum physicochemical properties. It also retards the release of drug, and it is a hydrophilic polymer. It is used as retardant, disintegrant, suspending agent, and matrix forming material. It is easily available and is quite economical. Being a natural polymer, it exhibits the property of biodegradation and mucoadhesion. Okra gum produces high viscosity mucilage at low concentrations. In continuation with the ongoing research on okra-based formulations, the major objective of the present investigation was to prepare, formulate and develop okra gum to be used as an EOR(Enhanced oil recovery) fluid.

II. DESCRIPTION

Okro (Abelmoschusesculentus (L.)Moench)isknown in many English-speaking countries as lady's fingers (Priyaet al., 2014). It belongs to family malvaceae and genus Abelmoschus .The geographical origin of okra is disputed, with supporters of South Asian, Ethiopian and West African origins.The plant is cultivated in tropical, subtropical and warm temperate regions around the world, National Research Council, "Okra". Lost Crops of Africa (2006). Okra can be grown on wide range of soils, but well drained fertile soils with adequate organic matter result to high yield (Akinyele, andTemikotan, 2007). The crop is widely cultivated throughout the year in the tropics. Okra is a nutritious vegetable which plays important role to meet the demand of vegetable which are scanty in the market Ahmedet al.(1995).

Okra plant or lady' finger was previously included in the genus Hibiscus, section Abelmoschus in the family Malvaceae(Linnaeus, 1753). The section Abelmoschus was subsequently

proposed to be raised to the rank of distinct genus (Medikus,1987). The wider use of Abelmoschus was subsequently accepted in the taxonomic and contemporary literature, Hochreutimer(1924).The genus Hibiscus by the characteristics of the calyx, spathulate, with five short teeth, connate to the corolla and caducous after flowering.Okra originated somewhere around the Ethiopia, and was cultivated by the ancient Egyptians by the 12th century B.C.

2.1STRUCTURE AND PHYSIOLOGY

Abelmoschusesculentus is cultivated throughout the tropical and warm temperate regions of the world for its fibrous fruits or pods containing round, white seeds. It is among the most heat and drought tolerant vegetable species in the world and will tolerate soils with heavy clay and intermittent moisture but frost can damage the pods. In cultivation, the seeds are soaked overnight prior to planting to a depth of 1-2 cm. Germination occurs between six days (soaked seeds) and three weeks. Seedlings require ample water. The seed pods rapidly become fibrous and woody, and to be edible, must be harvested within a week of the fruit having been pollinated. The fruits are harvested when immature and eaten as a vegetable Okra Seed.

2.2BIOCHEMICAL COMPOSITION OF OKRA

The composition of okra pods per 100 g edible portion is water 88.6 g, energy 144.00 kJ (36 kcal), protein 2.10 g, carbohydrate 8.20 g, fat 0.20 g, fibre 1.70 g, Ca 84.00 mg, P 90.00 mg, Fe 1.20 mg, β-carotene 185.00 µg, riboflavin 0.08mg, thiamin 0.04 mg, niacin 0.60 mg, ascorbic acid 47.00 mg. Protein, carbohydrate and vitamin C content of okra (Lamont, 1999), Saifullah(2009), Owolarafe.(2004), Gopalan(2007), and Dilruba (2009) and plays a vital role in human diet (Saifullah and Rabbani ,2009), (Kahlonet al., 2007). Consumption of young immature okra pods is important as fresh fruits, and it can be consumed in different forms Ndunguruand Rajabu(2014).

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

Okra fruit is principally consumed fresh or cooked and is a major source of vitamins A, B, C, minerals, Iron and Iodine and important vegetable source of viscous fiber but it is reportedly low in sodium saturated fat and cholesterol (Moawardet al., 1984), Kendall and Jenkins (2002),Adeboys and Oputa(1996). Presence of Fe, Zn, Mn and Ni also has been reported (Moyin-Jesu, 2007). Okra gives an invaluable source of vitamins, calcium, potassium and other mineral matters which are sometimes absent in the diet in developing countries. Seven days old fresh okra pods have the highest concentration of nutrients (Agbo. 2008)..

Carbohydrates are mainly present in the form of mucilage (Liuet al., 2005);Kumar (2009).The leaf buds and flowers are also edible (Doijode,2001). Okra seeds contain about 20% proteins and 20% oil Tindall(1093) and Charrier(1984). Okra seed oil has potential hypo-cholesterolemic effect (P.S. Rao and P.U.Rao ,1991). The potential for wide cultivation of okra for edible oil as well as for cake is very high (Rao,1985).The oil content of some varieties of the seed can be quite high, about 40%. Oil yields from okra crops are also high. A 2009 study found okra oil suitable for use as a biofuel(Farooq ,2010).

Fourier transform infrared spectroscopy (FT-IR) analysis is used for the identification of organic, inorganic and polymeric materials, by utilizing infrared light for scanning the samples (Deena et al., 2019). FT-IR analysis measures the range of wavelengths in the infrared region that are absorbed by a material. This is accomplished through the application of infrared radiation (IR) to samples of a material. The sample's ability to absorb the infrared light's energy at various wavelengths is measured to determine the material's molecular composition and structure. Unknown materials are identified by searching the IR spectrum against a database that has a wide range of reference spectra. Materials can be quantified using the FT-IR materials characterization technique as long as a standard curve of known concentrations of the component of interest can be created.Then, the signal is decoded by applying a mathematical technique known as Fourier transformation. This computer-generated process then produces a mapping of the spectral information. The resulting graph is the FT-IR spectrum which is then searched against reference libraries for identification.

The IR spectrum is a graph of infrared light absorbance by the substance on the vertical axis and the frequency (wavelength) on the horizontal axis.With the microscope attachment, samples as small as 20 microns can be analysed as well as quantifying contaminants or additives in materials.

SEM (Scanning electron microscopy) analysis helps us to identify the bonding structure of a sample. SEM relies on the detection of high energy electrons emitted from the surface of a sample after being exposed to a highly focused beam of electrons from an electron gun. This beam of electrons is focused to a small spot on the sample surface, using the SEM objective lens. (Laboratory Testing Inc. 2331 Topaz Drive, Hatfield, PA 19440)

Scanning Electron Microscopy uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. In most SEM applications, data is collected over a selected area of the surface of the sample and a two-dimensional image is generated that displays spatial variations in properties including chemical characterization, texture and orientation of materials. The SEM is also capable of performing analyses of selected point locations on the sample. This approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions, crystalline structure and crystal orientations.

The SEM gives the magnified images of the size, shape, composition, crystallography, and other physical and chemical properties of a specimen. The principle of the SEM was initially demonstrated during 1935 and 1939 by Knoll. Later, SEM was developed by von Ardenne. The

modern commercial SEM studies continued with extensive development in the 1950s and 1960s by Charles Oatley and his many students at the University of Cambridge..McHardy and Birnie gave historical developments along with its application to clay for SEM.

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that leave the material. XRD analysis enables the identification of different minerals with different crystal structures (Carteret al., 1986). In summary, the crystal X-ray diffraction phenomenon results from a scattering process in which X-rays are scattered by the electrons of atoms present in the sample without changing the wavelength.

III. PROBLEM STATEMENT

Low salinity polymer flooding has been conducted after low salinity environment was established by low salinity flooding (Alagic,

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

2010).In-situ saturation monitoring becomes a suitablw way to follow and measure the saturation change while flooding, as this gives vital information on how the flood front advances and informs about the possible in-situ redistribution of the fluids in the core. The concentration of the polymer affects the economy of the polymer flooding projects. Also the injectivity of polymer solution at higher concentrations is a crucial challenge for the polymer flooding design. After primary and secondary recovery energies are exhausted, about two third of OOIP are left behind in the reservoir. These can only be recovered through the EOR processes.

Primary recovery can recover from zero to over 50% of the original oil in place (OOIP), and the secondary recovery can recover from 30 to 50% of the original oil in place. Since oil production grows at a rate greater than reserve addition, there is a need to boost the reserve, and thislies with the application of tertiary recovery (EOR) which targets what is left(> 50 % OOIP).Most of the polymers used in the EOR applications are imported from other countries; as such it takes a lot of time. Also, the HPAM (partially hydrolyzed polyacrylamide) and biopolymer xanthan are susceptible to high temperature and their synthetic nature makes them harmful to the environment. (Agiet al.,2017). Hence there is need for the use of natural polymers.

In this research, extraction and characterization of okra (abelmoschusesculentus) sample were conducted.

3.1Aim and Objectives of the Study

3.1.1Aim

The aim of this research was to extract and characterise the okra (Abelmoschusesculentus), sample to be used in EOR applications.

3.1.2Objectives

(i)Okro sample collection and preparation
(ii)Extraction Procedure for Okro (Abelmoschusesculentus)
(iii)Physicochemical characterization of Okra mucilage

3.2Significance of the research

When a reservoir is flooded with polymer, the mobility ratio between the displaced fluid and the displacing fluid become favorable compared to the conventional water flooding. In the oil and gas industry, the synthetic polymer polyacrylamide in hydrolyzed form and the biopolymer xanthan are being used for this purpose. However, the polyacrylamide is susceptibleto high temperature and salinity. Also, its synthetic nature makes it harmful to the environment. The biopolymer xanthan has the problem of degradation and both are very expensive. With the shortfall in crude oil price and the high cost of exploitation and drilling new wells, there is need to look inward and think out of the box in formulating new improved polymers that can combat these problems. Natural polymers from agricultural and forest produce are abundant in nature, cheap and environmentally friendly. These agricultural and forest produce contain starch and cellulose which are known to have rigid and long polysaccharide chains that can withstand the harsh reservoir conditions (Augustine et al., 2018).

With the application of EOR using natural polymers additional oil will be recovered from the oil fields. Also this research work will result in economic boom to the oil and gas industries, job creation, and increase in the Nation's GDP.

IV. MATERIALS AND METHODS

4.1Okro sample collection and preparation

Okra (Abelmoschusesculentus) was obtained from local market of MudaLawal in Bauchi, Bauchi, State, Nigeria. Collected Okra was carefully washed and dried under shade for 24 h, and further dried in an oven at 30–40°C until constant weight was obtained. Okra size was pulverized through grinder. Powdered fruit was then passed through sieve no.#22 and stored in air tight container for further use. They were further dissolved with fresh water at 60℃ and stirred vigorously for proper dissolution. However, the dissolution was incomplete as there were undissolved particles still floating in the solution. These particles were carefully sieved out to obtain a fairly clear solution.

4.1.1Chemicals/reagents

Analytical grades of the following reagents were used in the course of this research. They include: sulphuric acid, petroleum ether, molish's reagent, sodium hydroxide, distilled water, pH indicator (methyl red and methyl blue) and hydrochloric acid.

4.1.2Apparatus and equipment

The following apparatus and equipment were used during the course of this study:
Laboratory oven, pH meter, viscometer, sieve no. #22, desiccator, soxhlet apparatus, round bottom flask, filter paper, kjeidahl flask, petri dish and volumetric flask.

4.2Methods

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

4.2.1Extraction Procedure for Abelmoschusesculentus

The extraction of mucilage was carried out according to the procedure by (Malviya, 2011; Gemedeet al., 2018) with little modification.

100 g of the powdered okra was dissolved in 300 ml of distilled water.This was heated and stirred continuously at 60°C for approximately 4h. The concentrated solution was carefully sieved and cooled at 6^{O}C.

Figure.1:Okro sample obtained local market of MudaLawal in Bauchi, Bauchi, State, Nigeria
A-Okro raw sample: B-Sliced okro sample: C- Pulverized okro sample.

4.3Physicochemical Characterization OfOkro Mucilage:

4.3.1pH

The pH was determined by the method as described by AOAC (2005). Two grams (2.0 g) of okra seed flour was poured into three beakers containing 20 ml of distilled water and allowed to stand for a while and an electric digital pH meter was used to determine the pH of the samples, Lala PK (1981). The pH meter was dipped into the sample and the reading was taken after about 4 min when it was stable.

The pH of the okro mucilage was determined in triplicate for statistical analysis to be more valid, and was found to be 7.5 as shown in the table below:

TABLE 1:pH

Sample	pH		
Okro	7.5	7.5	7.5

4.3.2 Bulk Density, g/cm^3

Accurately weighed amount of the dried okro sample (10 g) was taken and kept in a bulk density apparatus. The volume was noted. Hence, the bulk density was calculated using the formula:

$$Bulkdensity = \frac{Weightof\,powderedsample}{Bulkvolume} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

4.3.3Viscosity

The viscosity of Abelmoschusesculentus was determined by using Oswald's viscometer. The following equation(Lala, 1981)

$$S = W \times t_s\rho_s/t_w\rho_s \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

where, S=Viscosity of solution, W=Viscosity of water, t=time, and ρ=density.

4.3.4 Surface tension

It was determined by drop count method, using a stalagmometer according to the procedure of (Malviyaet al., 2010). Surface tension was calculated using the following equation:

$$\sigma_{solution} = \frac{\sigma_{water} \times m\ (solution)}{m\ (water)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots..(3)$$

Where,

$\sigma_{solution}$ = Surface tension of solution
σ_{water} = Surface tension of water
m (solution) = Weight of solution
m (water) = Weight of water

4.3.5Test for Carbohydrate

One hundred milligrams (100mg) of okro powder was dissolved in 5 ml of distilled water, and then filtered. 2 drops of alcoholic solution was added to 2 ml of the filtrate, and thoroughly shaken. 1 ml of concentrated sulphuricacid was slowly addedalong the sides of the test tube.

The violet color ring appeared at junction, showing presence of carbohydrates(Lala,1981).

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

TABLE 2: Test for Carbohydrate

Test	Observation	Inference
Okro sample solution + Molish's reagent + sulphuric acid.	violet color ring was observed at junction	Carbohydrate was confirmed.

4.4Proximate analysis

Proximate composition of moisture content, crude protein, ash content, and lipid content of the dried okra powdered samples were determined according to AOAC (2005). Protein was calculated from total nitrogen using the conversion factor 6.25. The percentage of total carbohydrate content of Abelmoschusesculentus seed flour sample was calculated by subtracting the percentage of moisture, ash, protein, and fats obtained from 100.

4.4.1 Moisture content

This was carried out after drying the okro sample under sun. The dried sample was taken and 1 g was weighed. After the weight was recorded, the sample was dried in a hot air oven at a temperature of 105°C. The weight was recorded at regular time intervals. This continued until a constant weighr was observed. The percentage of moisture content (Lala, 1981) was calculated using the equation:

$$Moisturecontent = \frac{Initialweig\ ht - Finalweig\ ht}{Initialweig\ ht} \times 100 \quad \ldots\ldots(4)$$

The okro sample was stored in an airtight container after its pulverization and sieving before constitution to be used as an EOR fluid

4.4.2 Ash content

Determination of ash content Pharmacological Assays of Plant-Based NaturalProducts (2016).The five grams (5g) of the powderedsample was weighed into a pre-weighed labelled crucible and placed inthe muffle furnace at a temperature of 450°C for 20 minutes. Thefurnace was allowed to cool before removing the crucible with itscontent. The crucible was later cooled in a desiccator and reweighed toget the ash content.. The result was calculated WHO; (1998) using the formula:

$$TotalAshvalue = \frac{weig\ htof\ heas\ h}{weig\ htof\ thegum} \times 100 \quad \ldots\ldots(5)$$

4.4.3 Lipid content

Determination of lipid content AOAC (2005): The five grams (5g)of powdered sample was put into the soxhlet extractor thimble wrapped with a filter paper and plugged tightly with cotton wool. 150 ml of petroleum ether (bpt 60-80°C) was poured into 300 ml round bottom flask containing anti-bombings and the soxhlet extractor assembly. The sample was extracted for 4h until the extract become colourless. The extract was poured into a dried pre-weighed beaker and the thimble rinsed with a little quantity of petroleum ether back into the beaker. The beaker was heated on a steam bath to drive off the solvent.The extracted fat left in the beaker was dried in a desiccator and weighed.

4.4.4 Crude fibre

Determination of crude fibre content: The five grams (5g)of powdered sample was put in a pre-weighed beaker. 50 ml of 1.25% H_2SO_4 solution was added and made up to 200 ml with distilled water and stirred. The mixture was heated with continuous stirring for thirty (30) minutes and allowed to cool and settle. Distilled water was added and allowed to settle, and then decanted.Decantation was repeated for six (6) times consecutively to make the mixture acid free. Fifty milliliter (50 ml) of 1.25% NaOH was added to 200 ml with distilled water in a beaker and heated for thirty minutes with continuous stirring. It was cooled and allowed to settle. Distilled water was added and decanted for six (6) times consecutively. The mixture was filtered with filter paper and kept for forty-five minutes for water to drain completely and the weight taken.

4.4.5Protein content

Determination of crude protein content (Modified Kjeldahl method): The analysis was carried out in three (3) stages, these were;

- The digestion stage.
- The distillation stage.
- The titration stage.

Digestion stage: 5 g of the powdered sample was weighed into a 250 ml kjeldahlflask,.then 2 g each of the kjeldahl catalysts (copper sulphate and sodium Sulphate) were weighed into the kjeldahl flasks. Anti-bumping granule was added and 30 ml of concentrated sulphuric acid was also added to the flask. The digestion flask was then placed on the heating

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

mantle for one hour before being transferred to electric stove. The digestion process proceeds with occasional swirling until a clear solution was obtained. The clear solution was transferred into a 100 ml standard flask and made up to the mark with distilled water.

Distillation stage: The 10 ml of the digest was measured into the micro distillation apparatus. 12.5 ml of 1.25% NaOH was also added to the flask. A condenser was connected from the distillation apparatus to a volumetric flask containing 10ml of 5% boric acid and 2 drops of double indicator (methyl red and methyl blue). The distillate was collected in a flask and then titrated with 0.1 ml standard hydrochloric acid until a pale pink colour end point was obtained

$$\%N = \frac{TD \times 0.0141 \times n \times V_a \times 100}{mass of sample \times V_d} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

where:

$TD =$ $Titre difference between Blank titer and sample titer$

$n = Normality of the acid used$

$V_a = aliquot volume of the distilled$

$V_d = Volume of digested sample$

$N = Nitrogen$ionic (%)

TABLE 3: Protein content

Titrations	1	2	3	Blank
Final Burette Reading (cm^3)	7.30	14.6	21.8	15.20
Initial Burette Reading (cm^3)	0.00	7.30	14.60	0.00
Vol. of acid used (cm^3)	7.30	7.30	7.20	15.20

4.5 Swelling Power

Swelling Power (SP) of powdered okro was determined using the method of (Babu andRamanathan 2014) with little modification as follows:

A 2 g (dry basis) sample was mixed with 180 ml of distilled water in a centrifuge tube and heated in a water bath at 50-90°C for 30 min with 10 ^{0}C interval. After heating, the suspension was centrifuged at 2200rpm for 15 min. The residue was drawn off by suction and dried for 4 hours at 120 ^{0}C in an oven and the weight of precipitated paste extracted from the sample was calculated. Swelling power was calculated as follows;

$$Swelling power = \frac{weig\,ht\ of\ precipitated\ \ paste}{Weig\,ht\ of\ Sample - weig\,ht\ of\ residue} \quad \ldots\ldots\ldots\ldots\ldots\ldots (7)$$

4.6 Water Solubility Index (%)

Water Solubility Index (WSI)

Two grams(2 g) of powdered okro sample was weighed into a 50-mL centrifuge tube and 30 mL ofdistilled water was added to it at 30^{O}C, and then stirred intermittently for 30 min. The solution was centrifuged for 10 min. The supernatant and pellet were carefully poured into a Petri dish, and allowed to dry overnight. Calculation was done using Eq.(8)

$$Water solubility index = \frac{weight of dried\ \ supernatant}{weight of powdered sample} X\ 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots (8)$$

4.7Fourier Transform Infra-red (FT-IR) Analysis

Fourier Transform Infrared Spectroscopy (FTIR) Analysis measures the infrared region of the electromagnetic radiation spectrum, which has a longer wavelength and a lower frequency than visible light. This spectrum is measurable in a sample when submitted to infrared radiation (IR). The basic theory at work is that the bonds between different elements absorb light at different frequencies.FTIR analysis was used to identify the presence of organic and inorganic compounds in the okrosample. Depending on the infrared absorption frequency range, 400-4000cm-1, the specific molecular groups prevailing in the sample was determined through spectrum data in the automated software of spectroscopy.

4.8Scanning Electron Microscopy (SEM) Analysis for okro powdered sample

The scanning electron microscopy (SEM) was performed on okro powdered sample to examine the physical structure change of sample using SEM model PhenomProX,byphenomWorldEinhoven. The Netherlands Sample was placed on double adhesive which was on a sample stub, was coated sputter coater by quorum technologies model Q150R,with 5nm of gold. Thereafter it was taken to the chamber of SEM machine where it was viewed via NaVCaM for focusing and little adjustment. It was then transferred to SEM mode, was focused and brightness contrasting was automatically adjusted,

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

afterward the morphologies of different magnification were stored in a USB stick.

4.9X-Ray Diffraction (XRD) Analysis

X-Ray Diffraction test was done to find out the orientation of the polymeric chain such as crystalline, microcrystalline or amorphous present in the okra sample. This X-ray diffraction measurement was performed using an X-ray diffractometer. A copper K-a X-ray source with 40kV voltage and 40mA power was used to record the diffraction patterns. The intensity as a function of the scattering angle is evaluated by the MS Excel spreadsheet tool after getting the diffraction patterns. The wavelength of the electron beam was Lambda, λ= 1.54063. The filter used here was a Solidstate detector

V. RESULTS AND DISCUSSION

.Result obtained in micromeritic characterization of AE (okro) mucilage are shown in Table 1.

TABLE 4: CHARACTERIZATION OF THE OKRO GUM

S/NO.	Paeameters	Report
1	Color	Light brown
2	Odor	Odorless
3	Taste	Tasteless
4	pH	7.5
5	Bulk density,	1.148 g/cm3
6	Viscosity	130cps
7	Surface Tension	0.041 joules/m^2
8	Carbohydrate	Present
9	Moisture content	4.00 %
10	Total Ash	11.50 %
11	Lipid content	1.20 %
12	Crude Fibre	11.50 %
13	Protein content	3.50 $
14	Swelling Power	0.775 g
15	Water Solubility Index	17.70 %
16	Loss on drying	9.917%

5.1Fourier Transform Infra-red (FT-IR) Analysis for okro sample

Okra powder is composed of organic substancesformed by functional groups such as polyphenoland carboxyl that serves as actives sites for wettability alterations. The identification of functional groupspresent in okra powder was performed by infrared spectroscopy. Fig. 2 shows FTIR spectrum of okrapowder in the resolution range of 4000-400 cm-1. In Figure 2, the larger band in the region of 3600–3100 cm^{-1} with a sharp peak centered at 3283.8cm^{-1} is characteristic O-H stretching vibration andhydrogen bond of the hydroxyl groups. Absorption peaks at 2926.0 and 2117.1 cm^{-1} correspond to C-Hstretching vibration from methyl and methylenegroup in cellulose and hemicellulose components; while at 1733.2 cm^{-1}refers to the carbonyl C=Ostretching vibration of unconjugated ketone,carboxylic acid and ester in lignin and hemicelluloses. The shoulder at 1606.5 cm^{-1} may be due to thepresence of water in the fibres. A little peak at 1401.5 cm^{-1} is associated to the angular deformation onthe OH bonding plane. The two peaks observed at 1364.2 $cm^{-1}$and 1315.8 cm^{-1} in the spectrum indicate the bending vibration of C-O and C-H groups ofthe aromatic ring in polysaccharides. Furthermore the absorption peaks around 1237.5 and 1013.8 cm^{-1}belong to polysaccharide in cellulose and indicatethe presence of C-O bonds associated with thepresence of functional groups such as alcohol, ether and ester (De Rosa et al., 2011; Jahanet al., 2015).

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

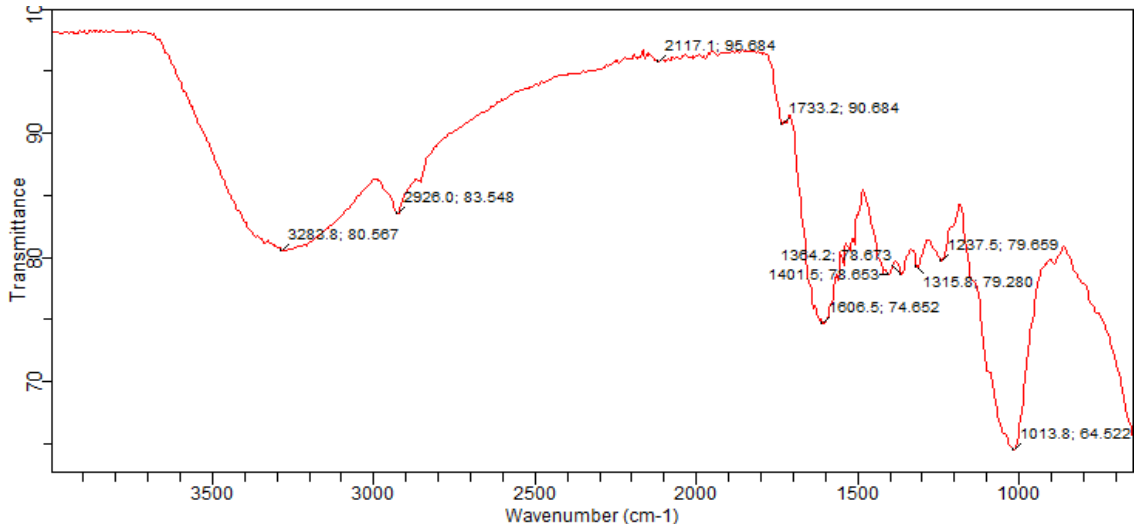

Figure.2: FT-IR spectrum of okrapowder

5.2Scanning Electron Microscopy (SEM) Analysis for okro powdered sample

The SEM image shows the uniform distribution of the pores which appeared to be heterogeneous in nature, and gives a well-defined appearance of the mesh (Figures. 3A , B, C, and D). Morphological analysis revealed that all particles had a glass structure and rough surfaces. Figure.3A shows the 300 time magnified structure of the particle while Figure.3B shows 1000time magnified structure.Figure.3 C and figure 3 D shows 1500, and 500 time magnified structure of theokro particle respectively.

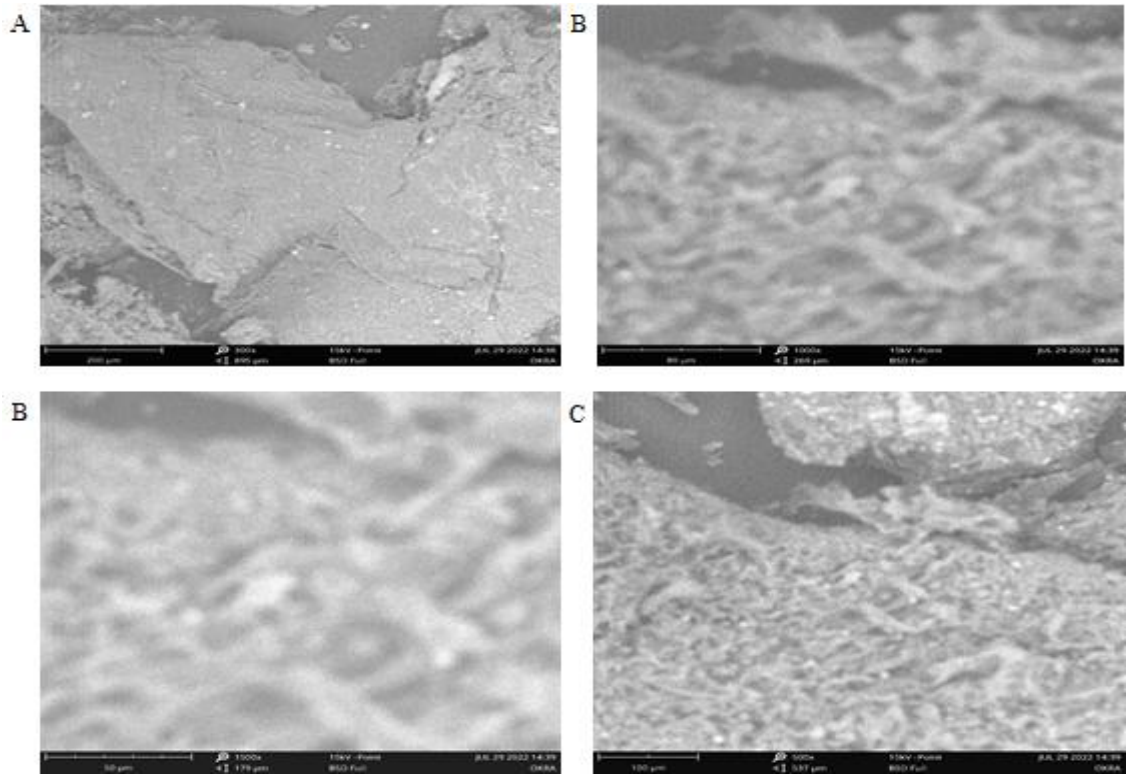

Figure3. SEM images of the 179-895µmokro intervals in powdered sample.

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

5.3XRD Analysis for okro sample

X-Ray diffraction (XRD) analysis was carried out using X-ray diffractometry to identify the amorphous nature of okro sample. The diffraction data for the okro sample were collected from 2 to 70^0 of 2θ starting from 0.02 at 1 seconds. The analysis was carried out to analyse the phase composition of the okro sample as shown in fig.4.The obtained pattern shows a mostly amorphous material including sylvine, brusite, lime, hopeite garnet, tridymite, oxammite, and halite with one crystalline phase which is the periclase (fig.5).

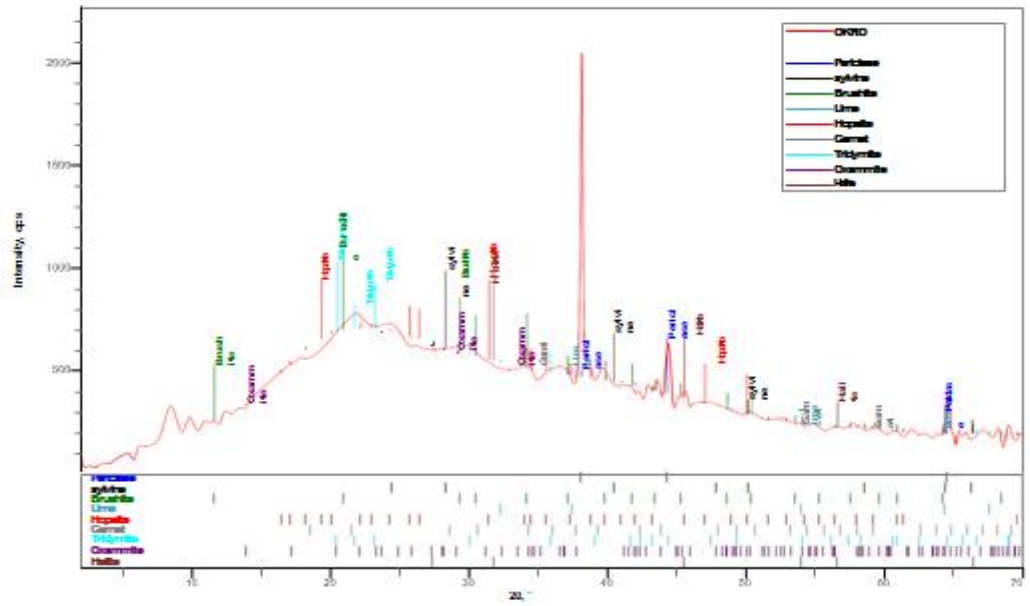

Fig.4: The XRD pattern for okro sample

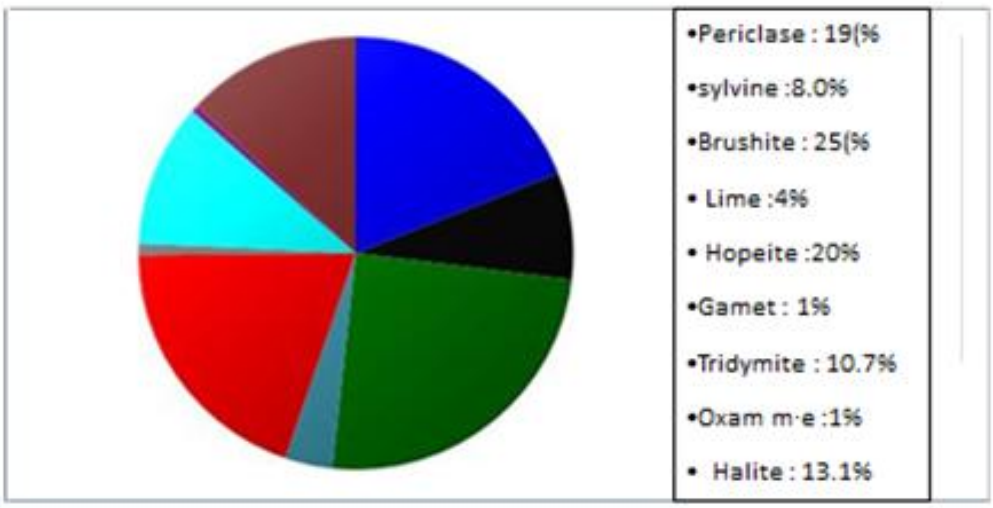

Fig.5: Piechart showing the mineralogical composition of okro sample

International Journal of Advances in Engineering and Management (IJAEM)
Volume 5, Issue 3 March 2023, pp: 1080-1093www.ijaem.net ISSN: 2395-5252

Table 5: Main mineralogical composition of okra sample

Mineral	Chemical formula	Concentration(weight %)
Periclase :	MgO	19
sylvine	KCl	8
Brushite	$CaH\ PO_4.2H_2O$	25
Lime	CaO	4
Hopeite	$Zn_3\ (PO_4)_2.4H_2O$	20
Garnet	$3(Ca,Fe,Mg)O.(Al,Fe)_2(SiO_4)_3$	1
Tridymite	SiO_2	10.7
Oxammite	$C_2H_8N_2O_4.H_2O$	1
Halite	NaCl	13.1

VI. CONCLUSION

Okro is a natural polymer obtained from the pods of okro plant. It has been extracted and used in the mucilaginous form. It is easily available and inexpensive. It is biodegradable, biocompatible, and also has swelling properties. Okra gum as a binder, and used as an EOR fluid. It has good physicochemical properties, i.e., hardness, friability, disintegration time, etc. The results obtained from this study established the fundamental characteristics of okra gum, and due to its swelling ability, it can be used successfully for EOR applications in the oil industry.

All the investigations were done for the characterization of okra fiber usingFT-IR, SEM andXRD analyses. FT-IR analysis revealed that okra powder is composed of organic substances formed by functional groups such as polyphenol and carboxyl that serves as actives sites for wettability alterations. The morphologies of okro powdered sample wass analyzed by SEM at high magnificationand the images are shown in Figure 3. From the Figure,The scanning electron micrographs(SEM) confirms the size andmorphology of okro powdered sample. The X-ray diffraction analysis confirmed the nature of Okra mucilage to be more amorphous thancrystalline. The obtained pattern on okro shows a mostly amorphous material including sylvine, brusite, lime, hopeite garnet, tridymite, oxammite, and halite with one crystalline phase which is the periclase.. The result showed that extracted okra mucilage exhibited good flow properties, the surface tension of 0.25% w/v solutions of mucilage was found to be 0.041 joule/m^2, total ash was 11.53% w/w, bulk density was 1.148 g/cm3,, moisture content was 4.00 %, lipid content was 1.2 %, crude fibrewas 11.50%, swelling power was 0.775 g, water solubility index was 17.70%, loss on drying was 9.917% and pH was found to be 7.5, and there was presence of carbohydrate. Extracted mucilage was soluble in warm water while insoluble in organic solvents. This showed that this can be safely used as an EOR agent without causing any adverse effect to the reservoir and its fluids.Okra mucilage was characterized for morphology, swelling, viscosity and flow properties. Polymerconcentration had the highest influence on microsphere size, entrapment efficiency and dissolution time while Okra ratio had the highest influence on swelling. Okra mucilage was a suitable polymer that could serve as an alternative to syntheticpolymers as an EOR fluid. Furthermore, increase in the concentrationof Okra mucilage in the formulations led to acorresponding increase in the size of the microspheresproduced. Higher concentrations of the Okra mucilageproduced more viscous polymer solutions which requiredmore energy to break into smaller droplets, thus resultingin the production of larger sized microspheres.

The following conclusions were drawn from this study:

- From the XRD analysis conducted on okro powdered sample, the obtained pattern shows a mostly amorphous material with few crystalline phases including sylvine, brusite, lime, hopeite garnet, tridymite, oxammite, and halite with one crystalline phase which is the periclaseAE-Okra mucilage has acceptable pH and organoleptic properties, so can be easily used to formulate natural polymer.
- The results showed that local polymer can be used as an alternative to the synthetic polymer under reservoir conditions.
- This mucilage is characterized by its high water absorbency and swell to form viscous substances due to its high content of polysaccharides
- It can therefore be concluded that AE-Okra gum is a naturalsemicrystalline polysaccharide, which is effective as an EOR fluid.

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