### Evaluation of the Frackability of Organic Shales Formations in Anambra Basin Nigeria from Triaxial Tests and XRD Results

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#### Abstract

The Ezeaku and Nkporo shales in the Anambra Basin are candidate formations for shale gas/oil development due to their high geochemical indices for generation and accumulation of hydrocarbons held in permeability jails. This study was carried out to determine the hydraulic fracturing potentials of these organic rich shales for exploration and development as unconventional reservoirs, using their mineralogy and strength parameters based on laboratory study of representative samples. Triaxial tests indicate that the shales derive their shear strength from both cohesion and internal frictional angles which fell between the values of 12 and 124 kN/m<sup>2</sup> and 3 to 11<sup>o</sup> respectively. Young's modulus values range from 75 kN/m<sup>2</sup> to 380 kN/m<sup>2</sup>. XRD diffractogram reflections of the shales indicate that the main clay mineral is kaolinite, with subsidiary illite, montmorillonite and chlorite. The non-clay mineral is mainly quartz, while feldspar and calcite were found in low quantities. Overall, clay minerals constituted approximately 39 % of the shales and the non-clay minerals made up 61%. The average mineral brittleness index determined from several empirical relationships fell between 0.30 and 0.91 with an average of 0.70 while the brittleness index obtained from the relationship between compressive strength and tensile strength fell between 0.27 and 0.86. Generally, high content of quartz increases the brittleness of shale, which in turn influences the initiation and propagation of artificially induced fractures. The overall implication of the results is that the shales are suitable for hydraulic fracturing.

Keywords: Anambra Basin, Evaluations, Frackability, Organic Shales, Triaxial Tests, XRD Results.

#### Introduction

In the upper 20 km of the earth's crust, 20% by volume of rocks are igneous and metamorphic rocks while sedimentary rocks constitute the remaining 80%. In general, 50% by volume of sedimentary rocks are sandstones and limestones, shales make up 40%. Shale is a fine-grained sedimentary rock composed of organic matter, clay and non-clay minerals from a variety of geological sources. It is abundant, highly fissile and is formed from compaction of about 50% particles of silt and clay sized mineral grains. In a conventional hydrocarbon systems, sandstones and carbonates are reservoirs while shale serve as source rock on account of their rich organic content. With developments in the oil industry, shale have assumed the roles of both source and reservoir for oil and gas, and are targeted as potential unconventional reservoirs (Vermylen, 2011; Ghassemi, 2012). Unconventional hydrocarbon resources are being developed in shale sequences globally by deploying hydraulic mechanisms to create artificial or enhance natural fractures through which hydrocarbons in the formations can be exploited. Identifying favorable shale formations, rich in hydrocarbons and easy to be fractured, is a major target for shale reservoir development.

In South Eastern Nigeria, Avbobo and Ogbe (1978) first brought into public attention, the occurrence of organic

rich shale which contain both oil and gas. Subsequently, Ekweozor and Udo 1988) and Ekweozor (1991) followed up on these reported occurrences to shed light on the geochemistry and occurrence of these oil and gas shales. Further studies on regional scale have been done by Ehinola and Abimbola (2002), Ehinola et al, (2004 and 2010), Unomah and Ekweozor (1993) all pointing to the occurrence of organically rich shale gas formations suitable for unconventional exploitation. This knowledge is supported by well documented data in terms of organic matter type and quality of the candidate geologic units. Geochemical analysis of Shale samples from Eze-Aku, Nkporo and Mamu Formations in the basin give total organic carbon (TOC) in the range of 1.9wt% to 3.32wt%, maximum temperature (Tmax) in the range of 424 °C to 467 °C, hydrogen index (HI) in the range of 306 to 600 mgHC/gTOC. These values imply rich hydrocarbon sources. An evaluation of the mineralogy and rock mechanical properties is crucial in determining if the shales are frackable to create some permeability (Grieser and Bray, 2007; Jacobi et al., 2009) and allow the contained fluid to flow from the natural permeability jail

Both mineral and elastic properties of the shales may be determined based on methods developed in the course of the exploration and development of shale gas unconventional reservoirs in China and North America where shales are extensively exploited as a result of innovations in technology. A rock's mechanical properties consist of the elastic (Poisson ratio, Young's modulus) and strength properties (unconfined compressive strength, internal angle of friction and cohesion). These properties affect the nature of response to applied stresses, and determines the frackability of shale formation (Evans *et al.* 1990; Ingram 1992; Jarvie 2008, Rickman 2008). Mineral composition is used to determine rock brittleness.

In this study, the authors have used laboratory testing of shale samples obtained at outcrops to determine these basic properties that determine the hydraulic fracturing potentials of the shales for exploration and development.

### **Study Area**

The project area lies between Longitudes 6°50' and 7°50' E and Latitudes 5° 40' and 6°15' N in Leru and Lokpanta Uturu and Afikpo areas in the southern the Anambra Basin (Fig. 1). These locations represent the best outcrop exposures of organic rich shale units. (Avbovbo and Ogbe,1978) based on the evidence of oil and gas shows and seepages that were recorded (Odigi, 2002, 2011, Nwajide 2005, Onyekuru and Iwuagwu 2010).

The lithostratigraphic packages in the Anambra Basin which was formed after the Santonian tectonism in Southern Benue Trough comprises a succession of four formations namely: the basal Nkporo Formation, overlain comfortably by Mamu Formation, Ajali Sandstone and Nsukka Formation (Petters, 1978; Agagu et al. 1985; Ojoh, 1992; Reijers, 1996). The pre-Santonian succession comprising the Asu-River Group, Eze-Aku shale and Awgu shale, were folded and faulted during the Santonian tectonic episode that was associated with magmatism. Afikpo is located in the Eastern flank of the Anambra Basin. Rocks in Afikpo area have been described in detail by Odigi and Amajor (2009), and consists of Albian, Turonian-Coniacian and Campanian-Maastrichtian sediments of Asu River Group and Eze-Aku Shales. Basaltic rocks intrude into the oldest sediments to form topographic highs.

### **Materials and Method**

### Samples and Sampling

The first stage of data acquisition for this work involved field sampling. The field work was carried out at best exposed outcrops of the extensively mapped shale

formations which have been discussed by some authors as being potentially hydrocarbon bearing (Whiteman, 1982.; Agagu et al. 1985, Petters and Ekweozor 1982, Petters, 1978 and 1986.; Akande and Erdtmann1998, Nwajide and Reijers 1996, Nwajide 2005, 2013) and organic facies and source rock characteristics (Ekweozor and Okoye,1980; Ekweozor and Unomah 1990; Akande et al. 1998; Akande et al, 1992a, 1992b, 2007, 2012; Ekweozor 2005; Akaegbobi et al. 2009; Ehinola et al. 2002, 2003, 2005., Obaje et al, 2004; Adeigbe and Salufu, 2010, Oluwajana and Ehinola 2016, 2018, 2020). Rock samples were obtained (Plate 1) for laboratory tests to obtain basic elastic parameters (especially the Youngs's modulus) and to analyse for the mineralogical composition of the shales. At each location, 5 different samples were obtained and a description of the lithology was undertaken according to the methods modified from Reijers (1996) and Tucker (1988). The coordinates of the sampled locations are shown in Table 3.1.

Shale mineralogy is an important indicator of its brittleness during hydraulic fracturing. The shale samples obtained during field study were preserved from the moment of retrieval in ziplock bags to avoid loss of pore water and strengthening, which could cause significant increase in strength and stiffness in both static and dynamic properties (Ghorbani et al., 2009).

Laboratory measurements were performed to determine the basic elastic parameters, especially the Youngs's modulus and mineralogical composition of the shales. Static elastic properties were determined from results of undrained unconsolidated triaxial tests performed in the laboratories of Bough Resources, Port Harcourt.

The samples study was subjected to unconsolidated undrained triaxial tests to obtain the stress-strain characteristic, and to determine static Young's modulus as well as the mineralogy of the shales. The test was performed in the laboratories of Bough Resources, Port Harcourt. Results of the test were used to determine the mode of failure, cohesion and internal frictional angles to validate the Mohr-Coulomb failure criterion. Data from triaxial tests were presented using Mohr diagrams. The normal and shear stress values at the time of failure were recorded and used as endpoints for semicircles on the Mohr diagram. The failure envelope was drawn as the tangent to all the circles. The circle with normal stress at zero and the tangent to the failure envelope gave the unconfined compressive strength, cohesion (or cohesive strength, the y intercept) and the tensile strength (negative x intercept) of the rock. The slope of the linearized envelope gives the coefficient of internal friction or the angle of internal friction. Also, data from the triaxial tests were used to construct graphs of stressstrain relationships from which the mechanical properties were obtained. Parameters obtained included compressive strength, tensile strength, Young's moduli, Poisson's ratios and the Mohr-Coulomb failure criterion which takes into account the cohesion and angles of internal friction of a material at failure. Unconfined compressive strength was taken as the peak stress. Young's modulus (E) was obtained from the slope of the axial stress versus axial strain curve.



Fig. 1: Geological Map of Anambra Basin with study area in red (modified from Nwajide and Reijers (1996).



**Plate 1.** Outcrops of shales in Anambra Basin (A). Nkporo shale at Owhutu (B). Nkporo shale at Leru (C). Ezeaku shale at Lokpanta (D). Ezeaku shale exposed at an erosional surface in Afikpo

	Location	Formation	Co-ordinates	Elevation (m)	Lithology Description
2	Amasiri – Akpoha road Afikpo Syncline	Eze-Aku Shale	05°54"26.0'N 07°54"23.3'E	15	Greyish Shale alternating with sandstone
3	College of Law, Gregory university Uturu	Eze-Aku Shale	05°51"65'N 07°52"83'E	11	Light grey non fissile Shale
4	Bore hole site at Gregory University Uturu	Nkporo Shale	05°50"56.7'N 07°25"40.3'E	112	Non fissile dark grey Shale
5	Leru junction	Nkporo Shale	05°54"46,2N 07°24"25.7'E	216.8	Highly fissile Shale
6	Leru section along Port Harcourt Enugu Expressway	Eze-Aku Shale	05°54"46.3'N 07°24"25.7'E	217.8	Highly fissile, dark grey Shale
7	Lokpanta	Eze-Aku Oil Shale	5°58"40.0'N 7°27"38.1'E	218.6	Highly fissile Shale

Table 1: Description of locations sampled during field study

In order to determine the whole rock mineralogy and percentage compositions of clay and non-clay minerals, ten shale samples were analysed using the XRD equipment in the Research Laboratories of the Nigeria Geological Survey Agency, NGSA in Kaduna. The laboratory uses an Empyrean diffractometer manufactured by Panalytical<sup>TM</sup>. The samples for the analysis were first air dried and pulverised. The pulverized samples were then deflocculated and dispersed using sodium hexametaphosphate. Following this the suspension was pipetted mounted on a slide. The samples were then analysed using the Empyrean diffractometer system fitted with a copper anode, and designed for rock and mineral analysis. The sample was analysed on the reflection-transmission spinner stage using the Theta-Theta settings. The diffractometer has an XRD range of  $4-75^{\circ}$  with a 2 $\theta$  step of 0.026261 at 8.67 seconds per step. A Programmable Divergent Slit with a 5mm Width Mask and the Gonio Scan was used. The diffractometer is equipped with an in-built diffraction software which produced the diffraction signatures. Phase identification was obtained by comparing the diffraction signal of a sample with a database of XRD mineral patterns. For example, for each diffractogram, the main peaks corresponding to highest diffraction intensities were identified and their  $2\theta$  values determined. These values were used to compare against the key lines and results assigned to the corresponding minerals. Files of d-spacings for hundreds of thousands of inorganic compounds are available from the International Centre for Diffraction Data (ICDD) as the Powder Diffraction File (PDF). The peaks obtained from these analyses were matched with the minerals phases from the ICDD database which is attached to the XRD. The XRD equipment is embedded with the processing software, XPert Highscore Plus which automatically performs this iteratively and identifies the minerals present in the whole rock samples. Results are commonly presented as peak positions at  $2\theta$  and X-ray

counts (intensity) in the form of a table.

The mineralogical indicator of brittleness was defined by the sum of clay minerals and quartz and other nonclay minerals obtained from XRD analysis. Rock brittleness, defined as the susceptibility of rocks to fracturing, is usually determined by its mineralogy – as quartz content (Jarvie 2008) or by the quartz content. Andreev (1995) reviewed about 20 different definitions of rock brittleness that use measurement methods based on any of strength, energy, fines content, penetration testing, point load testing, mineral composition and frictional angle. The method based on measurement of mineral composition is simple and was adopted in this study using the relationship found in Andreev (1995) expressed as

# $\mathbf{B} = \frac{Wqtz}{(Wqtz + Wcarb + Wclay)}$

Where  $W_{qtz}$ ,  $W_{carb}$  and  $W_{clay}$  are the content of quartz, clay and carbonate minerals, respectively.

Brittleness gives a measure of the susceptibility of rocks to fracturing. The higher the brittleness, the easier the rock fractures and the higher the shale frackability. Also, the triaxial test results were used to determine brittleness based on the following the method proposed by Goktan *et al.* (2005). The method proposed a relationship between compressive strength and tensile strength to calculate the brittleness index (BI) and rank the results. According to the method,

### $B_1 = \frac{\sigma c}{\sigma t}$

where  $\sigma c$  is the compressive strength, and  $\sigma t$  is the tensile strength. According to this method rock brittleness falls in four classes: high brittleness (BI>25), brittle (15<BI>25), medium brittleness (10<BI>15 and low brittleness (BI<10)

### **Results and Discussions**

The lithologic descriptions of the shales are summarized in Table1. Generally, shale at Lokpanta area belong to the Ezeaku Formation. It consists of dark blue marine shales. Evidence of trace fossils, including molds and casts are abundant at the outcrops. It is Turonian in age (Oloto, 2009), and was deposited during a transgression phase of the sedimentary cycle in Anambra Basin. The lithological associations of the Nkporo shales outcropping at Leru consist of light to dark grey shales and calcareous mudstones layers, and dark grey limestones. Thin beds of sandstones and sandy shale occur occasionally. The sand is very fine to medium grained, fairly well sorted and indurated. The shale shows very strong fissility. It has been interpreted as low energy offshore pro- deltaic, coarsening sequence of shale and shale/sand interbeds. Generally, the shales are dominated by organic rich black shales which are interlaminated with sand and clay which renders them discontinuous. Previous studies of the shales e.g. Ehinola and Abimbola (2002), Ehinola et al. (2004 and 2010), Unomah and Ekweozor (1993) indicate that the shales contain the most favourable TOC necessary for shale gas prospects. The presence of the laminations imply that the bulk permeability will increase which is in agreement with the observation by Jonk et al. (2010) that permeable sedimentary factors are important for fracture recharge which ultimately leads to good gas production in gas shales.

# Stress-Strain Relationships and Frackability Potentials

The failure mode of the shales was evaluated from the

shape of the cylindrical plug samples at the end of the triaxial tests. The triaxial test is a main method in the laboratory used to evaluate mechanical properties of rocks. When the samples were tested under quick unconfined unconsolidated conditions, the rocks failed by both shear and barreling. The Mohr failure envelopes (Fig. 2) clearly indicate that the shales derive their shear strength from both cohesion (or cohesive strength) and friction which fell between the values of 12-124 kN/m<sup>2</sup> and 3-11<sup>**0**</sup> respectively. These values indicate strong shales. Strong shales are more brittle and are more likely to initiate and propagate cracks during hydraulic fracturing.

Stress and strain data were used to generate graphs to gain insights into the deformation mode of the samples. The dependency of the stress-strain behaviour of the samples (Fig. 3) indicate that rocks usually show more brittle response at lower confining stress, and become more ductile as the stress increases (Meng et al. 2015). The stress- strain curves indicate brittle materials failing in a shear mode. The samples showed little strain and were closer to being linearly elastic before failure which confirms the brittle nature of the shales. By contrast, more ductile (or inelastic, or plastic) behaviour is indicated by the sample deforming in a non-elastic, (non-recoverable) manner before breaking. In contrast to brittle shales, ductile shale is a poor reservoir because such a reservoir will heal both natural and induced fracturing cracks. However, ductile shales serve as good seals and good fracture barriers for a reservoir. Brittle shale is more prone to natural fracture, and easily forms a complex network through hydraulic fracturing. The values of the derived Young's modulus, the ultimate tensile strength and yield stresses are shown in Table 2.



Fig. 2: Typical failure envelopes of shales at(a) Lokpanta (b) Leru

Principal Stress (kN/m<sup>2</sup>)

### **Shale Mineralogy and Brittleness**

Stress

The type o and quantity of clay and non-clay minerals that occur in shales is imperative in the design of fracture propagation (Laura (2015). The diffractogram reflections in Fig. 4a to 4d indicate that the main clay minerals and their species found in the shales, and their principal reflection peaks are

i. Kaolinite (7.2 Å)



Fig. 3: Representative stress-strain relationships of the shales (a) Lokpanta (b) Leru

Sample identification	Young's Modulus (kN/m²)	Ultimate Tensile Strength (kN/m²)	Yield Stress (kN/m <sup>2</sup> )	Undrained Cohesion (kN/m²)	Maximum deviatoric stress (kN/m <sup>2</sup> )	Axial Strain %	Brittleness index	
L3 S2	12.00	380	360	188	375	5.6	0.79	
L4 S2	83,33	310	300	150	310	5.6	0.86	
L5 S1	90.83	400	390	200	401	8.8	0.41	
L5 S2	84.62	325	320	150	325	4.4	0.46	
L5 S3	91.67	153	150	75	153	5.2	0.46	
L7 S1	380.00	106	102	53	106	4.0	0.50	
L7 S3	12.50	48	44	24	224	6.0	0.33	
L7 S6	10.00	31.5	29	16	31	4.8	0.38	
L7 S19	113.00	223	110	224	8.8		0.27	
AF L3	75.00	180	179	90	179	4,8	0,42	

 Table 2: Strength indicators and brittleness index of the shales

- ii. Montmorrilonite (10-15Å)
- iii. Illite(10Å)
- iv. Chlorite (7 Å)

The non-clay minerals were

- i. Quartz.
- ii. Feldspars (albite).
- iii. Calcite carbonate
- iv. iron bearing minerals hematite.

This mineral composition is consistent with Cretaceous shales. Other than quart, the non-clay minerals occur in very small amounts. The complex assemblage of clay minerals in agreement with results obtained by Ukaonu (2009) who studied the mineralogy of Upper Cretaceous Formations in the Anambra Basin and shale samples from Nkoro Formation and XRD analysis yielded mainly the clay minerals kaolinite, smectite and illite. Oti (1990) obtained similar clay mineral phases in addition to interstratified chlorite-smectite and micavermiculite. In addition to these minerals, Akpokodje (unpublished diffractograms) obtained smectite-illite interlayered clays in Nkporo Formation. The presence of illite which is the most thermally stable clay mineral often seen in gas shales is in agreement with the findings of Josh et al. (2012)

This mineral composition is consistent with Cretaceous shales. Other than quart, the non-clay minerals occur in very small amounts. The complex assemblage of clay minerals is in agreement with results obtained by Oti (1990) whose study in the basin showed the occurrence of five principal clay mineral phases including interstratified chlorite-smectite and mica-vermiculite, chlorite, illite and kaolinite. Ukaonu (2009) Nkporo Formation and XRD analysis yielded mainly the clay minerals kaolinite, smectite and illite. In addition to these minerals, Akpokodje (unpublished diffractograms) obtained smectite-illite interlayered clays in Nkporo Formation. The presence of illite in the samples which is the most thermally stable clay mineral often seen in gas shales is in agreement with the findings of Josh et al. 2012).

the shales is summarized in Table 2. It shows that clay mineral composition varies from 1 to 57 %. The quartz content fell between 8 and 32 % with an average of 26.82%. The carbonate mineral calcite occurs in only one sample. The feldspars, albite occur in subsidiary amounts (10.25 %) in only two out of all the samples analysed. Overall, the clay minerals make up 39.61 %, while the non-clay minerals constitute 59.69 % by composition (Fig. 5).



Fig. 4c: Diffractogram of Nkporo shales at 1440 ft (439 m) in a borehole

Semi-quantitative estimate of the overall mineralogy of



Table 3: XRD derived mineral species composition of the shales by %

Clay Minerals Non clay minerals								Brittleness Index							
Sample No.	Sample ID	Formation	Montmorrilonite	Kaolinite	Illite	Chlorite	Quartz	Albite	Calcite	Gypsum	Jarosite	Haematite	Andreev (1995)	Javier et al.(2007)	Jin (2014)
1	NK 1	NK	-	24.00	<u> -</u>	523	76.00	-	-	-	9	-	0,76	0.76	0.76
2	NK 2	NK	-	19.00	-	8 <b>-</b> 9	60.00	21.00	-	-	-	875	0.63	0.60	0.81
3	NK 3	NK	2	11.00	<u> -</u>	828	56.00	28.00	-	-	5.00	-	0.83	0.56	0.89
4	EZK 1	EZK	-	9.00	-	5 <del></del> 8	67.00	-	24.00	-	-	3 <b>7</b> 3	0.67	0.67	0.91
5	EZK 2	EZK	2,00	32.00	<u>_</u>	12	62.00	-	-	2	<u>.</u>	4.00	0.65	0.62	0.62
6	EZK 3	EZK	-	12.90	57,40	1.00	29.70		-	Ħ	-	5 <b>5</b> 5	0.30	0.30	0.87
7	NH 1	NK	2	13.00	-	121	79.00	<u> </u>	-	2	8.00	( <b>1</b> )	0.87	0.79	0.87
8	NH 2	NK	1.00	21.00	-	-	76.00	-	-	2,00	-		0.78	0.76	0.86
9	AL 1	NK	1.00	-	2	36.40	62.60	2	2	2	2	-	0.62	0.62	0.62
10	AL 2	EZ	-	27.00	-		73.00	-	-	-	-	-	0.73	0.73	0.73
	Minimum	L .	1.00	9.00	57.40	36.40	29.70	21.00	24.00	2.00	5.00	4.00	0,30	0.30	0.62
	Maximum	ı	2.00	32.00	57.40	36.40	79.00	28.00	24.00	2.00	8.00	4.00	0.87	0.79	0.91

NK1 = Nkporo shale at Leru NK 2 = Nkporo shale at Leru

NK 3 = Nkporo shale at Uturu (3,600ft)

EZK 1 = Ezeaku shale at Lokpanta

EZK 2 = Ezeaku shale at Amasiri EZK 3 = Ezeaku shale at Uturu NH 1 = Nkporo shale at 1080 ft

**NH 2** = Nkporo shale at 1480ft

Al 1 = Nkporo shale at 2290 ft

Al 2 = Nkporo shale at 2354 ft

Clay minerals, 39.31%
Non-clay minerals, 59.69%

Fig. 5: Mineralogical composition of the shales

Clay minerals primarily form from the weathering process. The nature of clay formed during the weathering process depends upon three factors: mineralogical and textural composition of the parent rock, composition of the aqueous solution and the rate of fluid flow and pore network. Their formation has been described by Deer et *al.* (1975) and Borchardt (1977). Kaolinitic clay minerals form primarily during hydrothermal alteration or weathering of feldspars under acidic condition. The smectites on the other hand commonly result from the weathering of basic rocks by crystallization from solution high in soluble silica and magnesium Their formation is favoured by level to

gently sloping topography in mildly alkaline environments that are poorly drained in order to retain ions (e.g. Mg) freed in the alteration. Illites form through the alteration of other clay minerals, and during the degradation of muscovite where K<sup>+</sup> replaces some of the interlayer cations of montmorillonites or vermiculites, and heat and pressure cause the dehydration and collapse of the clays into non-expanded forms. Chlorite minerals are common components of low-grade greenschist facies metamorphic rocks, and of igneous rocks as hydrothermal alteration products of ferromanganese minerals. Mixed-layer clays can form by a variety of processes: weathering involving the removal or uptake of cations (e.g. K), hydrothermal alteration, or removal of hydroxide interlayers. As products of burial diagenesis, they are useful indicators of burial of sediments. In some cases, their formation may occur in an intermediate stage in the formation of swelling minerals from non-swelling minerals or vice versa (MacEwan and Ruiz-Amil, 1975).

X-Ray diffraction confirm that the outcrop samples are comprised dominantly of minerals with transportation and deposition of argillaceous sediments along with siliciclastic controlled by tectonics and climate in the depositional history of the Anambra basin. The samples would belong to the basic volcanic and intermediate intrusive rich in feldspars derived from the weathering of rocks in Abakiliki anticlinorium the provenance of sedimentary fill in the Anambra Basin.

Altogether, clay mineralogy, quartz content and feldspars favour brittleness. The mineralogy suggests that theoretically, the shales are brittle enough to initiate microfractures and keep them open. The low amount of the smectites which have high hydro affinity, will therefore not limit the injection of fluids during hydraulic fracturing in these source-reservoir rock shales. The occurrence of quartz alongside with the clay minerals will contribute to the brittleness of the shale which and subsequently, its ease of frackability. The higher the brittleness, the easier the ability of rocks to develop micro-factures and the higher the frackability of the shales. Meng et al. (2015) stated that brittleness is an important characteristic of rocks because it influences very strongly the failure process of rocks resulting in their breakage. It also controls rock failure when they are subjected to stress during loading. The strength properties of rocks shown in Table 2 are composite factors that together with mineralogy, determine the likelihood or otherwise of frackability as well as the energy required to create an effective fracture network in shales. In contrast, to brittle shales, ductile shales are a poor reservoir because such a reservoir will heal both natural and induced fracturing cracks. However, ductile shale serves as a good seal and a good fracture barrier for a reservoir. Brittle shale is more prone to natural fracture, and easily forms a complex network through hydraulic fracturing. Therefore, it is necessary to quantify the brittleness of shale from its mineralogical composition from XRD data.

Generally, high content of quartz increases the brittleness of shale, which in turn influences the initiation and propagation of artificial fractures.

A ternary diagram of the mineralogy of the shales, plotted using the Britt and Schoeffler (2009) tri-plot is shown in Fig. 5 with plotted data derived from the present study. The majority of the samples plot to the left of the line which represent 40 % clay mineral constituents. Britt and Schoeffler (2009) consider shales with clay constituent in excess of 40 % too high to be considered 'widely prospective' using a mineralogical analysis of prospective shale plays in North America which are mainly silica and carbonate materials with few clay constituents. In addition to the optimal clay minerals content (approximately 39%), the position of the shales which fall mostly to the left on the ternary diagram imply that the shales will present little challenges in maintaining fractures open during production of the gas that is held in permeability jail during hydraulic fracturing. Although the mineral make-up of shales influence (and control, to a large extent) the brittleness and fracabiliy of the shales the reverse is not necessarily true or valid. For example, shales with 'too much clay' exceeding 40 % are still prospective e.g. the Haynesville clay and the wellknown Marcellus shale which can have mineralogy of up to 50 %, are among the top shale gas producing formations in the US (Josh et al. 2012). In this instance, the fracture stimulation design is modified where higher gelled proppant concentrations are used to obtain very good results. To achieve this in shales with clay minerals in excess of 40 % which are considered to be brittle, the stimulation of fractures is employed using clean, nondamaging fissure dilating fluid like water commonly referred to as water fracs. Gao (2015) obtained mineralogical compositions of shales in Ordos Basin, China, similar to the shales in this study and concluded that the shales will perform optimally during production. It is expected that the shales in Anambra Basin that contain about 39 % of clay minerals which is within the cut off of Britt and Schoeffler (2009) will be prospective.



Fig. 6: Tri-plot of the mineral composition of the shales. Majority of the samples plot to the left of the 40 % clay line

Brittleness is one of the most important mechanical properties of rock that controls artificial fracking. Andreev (1995) reviewed about 20 different definitions of rock brittleness that use measurement methods based on any of strength, energy, fines content, penetration testing, point load testing, mineral composition and frictional angle. The methods based on measurement of mineral composition (Andreev 1995, Jarvie *et al.* 2007, Jin *et al.* 2014) are simple and were adopted in this study. The brittleness relationships according to these methods and the results obtained are summarized in Table 3.

Table 3: Brittleness index using different methods.

Method	Brittleness	Min	Max	Average	
Andreev, 1995	B = Wqtz	0.30	0.87	0.68	
Javia at al 2007	(Wqtz + Wcarb + Wclay Wqtz	0.30	0.79	0.64	
Javie el ul, 2007	$B = \frac{W \text{ tot}}{W \text{ tot}}$ WQFM +Wcarb	0.50	0.75	0.04	
Jin, 2014	$B = \frac{W}{W}$ tot	0.62	0.91	0.79	

Where

 $W_{qtz}$  is content of quartz, Wcarb is content of clay minerals,  $W_{clay}$  is content of carbonate minerals and WQFM is sum of Quartz, feldspar and mica content

According to Jin *et al.* (2014) a formation with a brittleness of 1.0 is the best candidate for hydraulic fracturing candidate, and formation with brittleness of 0 is the worst candidate. The average brittleness index values fell between 0.30 and 0.91 with 0.70 average. The average brittleness coupled with brittle mineral composition and sand lamination make the shales suitable for fracking.

The presence of clay minerals and quartz is an important brittleness indicator. According to Meng *et al.* (2014), the most understandable and acceptable definition of brittleness may be that rock terminates by fracture at or only slightly beyond the yield stress (i.e., little or no plastic deformation occurs at failure. Brittle failure of rock under high stress frequently occurs in the deep subsurface due to complex geological and stress conditions. It is therefore imperative to evaluate brittle failure intensity of the surrounding rock under certain stress states during any engineering project, including the design and operation of hydraulic fracturing programmes to poduce oil or gas from shales. It is well known that minerals are the fundamental parts influencing the brittleness of rock (Jarvie *et al.* 2007, Slatt and Abousleiman 2011). Early methods of determining brittleness equated weight fraction of quartz with mineralogical brittleness (Jarvie *et al.* 2007). Later, the fraction of dolomite was added to quartz when it was observed that dolomite tends to increase the brittleness of shales (Wang and Gale 2009). Similarly, silicate minerals such as feldspar and mica, as well as other carbonate minerals like calcite in dolomite, tend to be more brittle than clay in shale reservoirs. The Jin (2014) expression of brittleness was proposed to include all these minerals as an improvement over the other expressions in Table 3

Another favourable complementary factor is the occurrence of sand laminations significantly throughout these shale formations. This lithology enhances horizontal permeability. A combination of these mineralogical factors is known to improve the shale reservoir performance greatly by acting as proppants to arrest the self-healing of the cracks, instead keeping them open. Therefore, on the basis of the mineral composition, the shales in this study which consists of highly brittle minerals will generate natural cracks. Shale reservoir respond better to the hydraulic fracturing process when they contain high quantities of

Adeigbe, O.C. and Salufu, A.E. (2010). Geology and petroleum potential of Campano-Maastrichtian sediments in the Anambra Basin, SE Nigeria. *NAPE Bulletin*, 22(1), 13-17.

Agagu, O.K., Fayose, E.A. and Petters S.W. (1985). Stratigraphy and sedimentation in Anambra Basin. *Journal of Mining and Geology* 22, 25-36

- Akaegbobi, I.M, Amechi, G.C. and Boboye, O.A. (2009). Source Rock Potential and Depositional Environment of the Campanian-Maastrichtian Shales outcropping along the Enugu-Leru axis, Anambra Basin, Nigeria. *Nigerian Association* of Petroleum Explorationist Bulletin, 21, 25-37
- Akande, S.O., Egenhoff, S.O., Obaje, N.G., Ojo, O.J., Adekeye, O.A. and Erdtmann, B.D. (2012). Hydrocarbon potential of Cretaceous sediments in the Lower and Middle Benue Trough, Nigeria: Insights from new source rock facies evaluation: *Journal of African Earth Sciences*, 64, 34-47.
- Akande, S.O., Hoffknecht, A. and Erdtmann, B.D. (1992). Rank and petrographic composition of selected Upper Cretaceous and Tertiary coals of southern Nigeria. *International Journal of Coal Geology*. 20, 209–224.

brittle minerals. From the forgoing discussion, the shales in this study are frackable

### Conclusion

This study was carried out to evaluate the frackability of the reported gas shales in the Anambra Basin based on triaxial test results and XRD derived mineralogy. The shales derive their strength from cohesion and angles of internal frictional resistance. The shales are composed of 39 % clay minerals and 61 % non-clay minerals. Brittleness index determined from the strength and mineralogy of the shales and fell between 0.30 and 0.9. It is therefore concluded that the shales in the Anambra Basin will be frackable during hydraulic fracturing in order to produce the hydrocarbon contain in them. However more matured sections of the shales require more studies to gain a better understanding of the pore architecture of the shale and factors that control them.

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### References

- Akande, S.O. and Erdtmann, B.D. (1998). Burial metamorphism (Thermal Maturation) in Cretaceous Sediments of the Southern Benue Trough and the Anambra Basin, Nigeria. *AAPG* 82, 1191–1206
- Akande, S.O., Ogunmoyero, I.B., Petersen, H.I. and Nytoft, H.P. (2007). Source Rock Evaluation of Coals from the Lower Maastrichtian Mamu formation, South Eastern Nigeria. *Journal of Petroleum Geology*, 30, 303–324
- Andreev, G.E. (1995). Brittle failure of rock materials: test results and constitutive models. A. A. Balkema, Rotterdam
- Britt, L.R. and Schoeffler J. (2009). The Geomechanics Of A Shale Play: What Makes A Shale Prospective. Society of Petroleum Engineers (SPE) Eastern Regional Meeting, 23-25 September, Charleston, West Virginia, USA. doi: 10.2118/125525-MS 9p
- Ehinola, O.A. and Abimbola, A.F. (2002). Preliminary assessment of major and trace elements content in the middle Cretaceous black shales of the Abakaliki fold belt, Southeastern Nigeria. *Nafta*, 53(9), 323-326.

- Ehinola, O.A., Bassey, C.E. and Ekweozor, C.M. (2003). Preliminary Studies of the lithostratigraphy and Depositional Environment of Oil Shale Deposit of Abakaliki Anticlinorium South Eastern Nigeria. *Journal of Mining and Geology*, 39, 2, 85-94
- Ehinola, O.A., Sonibare, O.O., Falode, O.A. and Awofala, B.O. (2005). Hydrocarbon potential and thermal maturity of Nkporo Shale from Lower Benue Trough, Nigeria. *J. Appl. Sci.* 5, 689–
- Ekweozor, C.M. (2005). Searching for petroleum in the Anambra basin, Nigeria. *Hydrocarbon Potentials* of the Anambra Basin: Geology, Geochemistry and Geohistory Perspectives, Great AP Express Publication Limited, Nsukka, 83-110.
- Ekweozor, C.M. and Okoye, N.V. (1980). Petroleum Source-Bed Evaluation of Tertiary Niger Delta: Geologic Notes. *AAPG Bulletin*, 64(8), 1251-1259.
- Ekweozor, C.M. and Udo, O.T. (1988). The oleananes: origin, maturation and limits of occurrence in Southern Nigeria sedimentary basins. In: Organic Geochemistry in Petroleum Exploration, Pergamon.
- Ekweozor, C.M. and Unomah, G.I. (1990). First Discovery of Oil Shale in the Benue Trough, Nigeria. *Fuel* 69, 502–508.
- Evans, M.A., DeLisle, A., Leo, J. and Lafonte, C.J. (2014). Deformation conditions for fracturing in the Middle Devonian sequence of the central Appalachians during the Late Paleozoic Alleghenian orogeny. *AAPG Bulletin*, *98*(11), 2263-2299.
- Gao, R.X., Wang, X. and Feng, J.F. (2015). Fracability evaluation of lacustrine shale in the Yanchang Formation of Southeastern Ordos Basin. *Energy exploration & Exploitation* 33 (3) 363–37
- Ghassemi, A. (2012). A Review of Some Rock Mechanics Issues in Geothermal Reservoir Development. <u>Geotechnical and Geological</u> <u>Engineering</u> 30, 647–66
- Ghorbani, A., Zamora, M. and Cosenza, P. (2009). Effects of Desiccation on the Elastic Wave Velocities of Clay-Rocks. *International Journal of Rock Mechanics and Mineral Science* 46, 1267–1272
- Goktan, R.M. and Yilmaz N.G. (2005). A new methodology for the analysis of the relationship between rock brittleness index and drag pick cutting efficiency. *The Journal of the South African Institute of Mining and Metallurgy* 105(10), 727733.

- Grieser, W.V. and Bray, J.M. (2007). Identification of production potential in unconventional reservoirs. In *Production and Operations Symposium*. Society of Petroleum Engineers.
- Jacobi, D.J., Breig, J.J., LeCompte, B., Kopal, M., Hursan, G., Mendez, F.E., ... & Longo, J. (2009). Effective geochemical and geomechanical characterization of shale gas reservoirs from the wellbore environment: Caney and the Woodford shale. In SPE annual technical conference and exhibition. Society of Petroleum Engineers.
- Jarvie D.M. (2008): Unconventional shale resource plays: shale-gas and shale-oil. opportunities. Fort Worth Business Press Meeting, June 19 2008. (shaledigest.com/documents/dan\_jarvie. pdf) (access: May 2014).
- Jarvie, D.M., Hill, R.J., Ruble, T.E. and Pollastro, R.M. (2007). Unconventional shale-gas systems: The Mississippian Barnett Shale of north-central Texas as one model for thermogenic shale-gas assessment: *AAPG Bulletin*, 91(4), 475–499, doi:10.1306/12190606068
- Jin, X., Shah, S.N. and Roegiers, J.C. (2014). Fracability Evaluation in Shale Reservoirs-An Integrated Petrophysics and Geomechanics Approach. SPE-168589 SPE Hydraulic Fracturing Technology Conference held in The Woodlands, TX. Feb. 4-6
- Joint Committee on Powder Diffraction Standards, JCPDS (1980). Mineral Powder Diffraction File I and II USA. International Centre for Diffraction Data 1-24
- Josh, M., Esteban, L. Piane, C.D. and Sarout, J. (2012). Laboratory characterisation of shale properties. *Journal of Petroleum Science and Engineering* 88–89:107–124 ·DOI: 1
- Laura, A. (2005). Geomechanical Properties of Shale Rock from Baltic Basin in Poland Area. Agh Drilling, *Oil & Gas* 32(2): 369-279. http://dx.doi.org/10.7494/drill.2015.32.2.369
- Meng F., Zhou, H., Zhang, C., Xu, R. and Lu, J. (2015). Evaluation Methodology of Brittleness of Rock Based on Post-Peak Stress–Strain Curves. *Rock mechanics and Rock Engineering* 48: 1787-1805
- Nwajide, C.S. and Reijers, T.J.A. (1996). The geology of Southern Anambra Basin. In: Selected Chapters on Geology, Sedimentary Geology and Sequence Stratigraphy in Nigeria and Three Case Studies and a field Guide (ed) T.J.A. Reijers, Shell Petroleum Development Company of Nigeria Corporate Reprographic Services, Warri; p. 133-148

- Nwajide, C.S. (2005). Anambra Basin of Nigeria: Synoptic Basin Analysis as a Basis for Evaluating its Hydrocarbon Prospectivity. In (ed) C. O. Okogbue: Hydrocarbon Potentials of the Anambra Basin; Geology, Geochemistry and Geohistory Perspectives. Proceedings of the first Seminar organized by the Petroleum Technology Development Fund (PTDF) Chair in Geology, University of Nsukka. P. 1-46
- Nwajide, C.S. (2013). Geology of Nigeria's Sedimentary Basins. CSS Bookshops Limited. Lagos. 565p.
- Obaje, N.G., Wehner, H., Scheeder, G., Abubakar, M.B. and Jauro, A. (2004). Hydrocarbon prospectivity of Nigerian inland basins: from the viewpoint of organic geochemistry and organic petrology. AAPG Bull. 88, 325–353.
- Odigi, M.I. (2002). Petrography, diagenesis and reservoir development of sandstones in the Asu River Group, Lower Benue Trough, southeastern Nigeria. *Journal of Applied Sciences and Environmental Management*, 6, 34-38.
- Odigi, M.I. (2011). Diagenesis and reservoir quality of Cretaceous Sandstones of Nkporo Formation (Campanian) southeastern Benue Trough, Nigeria. *Journal of Geology and Mining Research*, 3(10), 265-280.
- Odigi, M. and Amajor, L.C. (2009). Brittle deformation in the Afikpo Basin (Southeast Nigeria): Evidence for a terminal Cretaceous extensional regime in the Lower Benue Trough, Nigeria. C h i n . J . G e o c h e m . 2 8 , 3 6 9 https://doi.org/10.1007/s11631-009-0369-2
- Ojoh, K.A. (1992). The southern part of the Benue Trough (Nigeria) Cretaceous stratigraphy, basin analysis, paleo-oceanography and geodynamic evolution in the Equatorial domain of the South Atlantic. *NAPE Bulletin*, 7(2), 67-74.
- Oloto, I.N. (2009). Palynology and Sequence stratigraphy: case study from Nigeria. Legacy Integrations Nigeria Ltd. Port Harcourt.254p.
- Oluwajana, O.A. and Ehinola, O.A. (2016). Hydrocarbon Charge Modelling of Anambra Basin, South Eastern Nigeria: Implications for Cretaceous Sourced Plays. Arabia journal of Geoscience.

- Onyekuru, S.O. and Iwuagwu, C.J. (2010). Depositional environments and sequence stratigraphic interpretation of the Campano-Maastrichtian Nkporo shale group and Mamu Formation exposures at Leru-Okigwe axis, Anambra Basin, Southeastern Nigeria. *Australian Journal of Basic and Applied Sciences*, 4(12), 6623-6640.
- Petters, S.W. (1978a). Stratigraphic evolution of the Benue Trough and its implications for the Upper Cretaceous paleogeography of West Africa. J. Geol. 86, 311–322.
- Petters. S.W. and Ekweozor. C.M. (1982). Petroleum geology of Benue Trough and southeastern Chad Basin, Nigeria. AAPG Bulletin, 66, 1141-1149.
- Reijers, T.J.A. (1996) Selected chapters in Geology, Sedimentary Geology and Sequence Stratigraphy in Nigeria and three case studies and a Field Guide SPDC Reprographic services, Warri. 197.
- Rickman, R.M., Mullem, E.P., Grieser, B. and Kundert, D. (2008). A practical use of shale petrophysics for stimulation design optimization: All shale plays are not clones of the Barnett shale: SPE Annual Technical Conference and Exhibition, Denver, Colorado, SPE-115258-MS, 11 p., doi:10.2118/115258-MS.
- <u>Sayers</u>, C.M. (2005). Seismic anisotropy of shales. *EAGE Geophysical Prospecting* 53(5), 667-676 <u>https://doi.org/10.1111/j.1365</u>-<u>2478.2005.00495.x</u>
- Unomah, G.I. and Ekweozor, C.M. (1993). Petroleum source-rock assessment of the Campanian Nkporo shale lower Benue Trough, Nigeria. *NAPE Bull.*, 8(02), 172-186.
- Vermylen, J. and Zoback, M. (2011). Hydraulic Fracturing, Microseismic Magnitudes, and Stress Evolution in the Barnett Shale, Texas, USA. Journal Soc. Of Pet. Eng. DOI: <u>10.2118/140507-MS</u>
- Whiteman, A. (1982). Nigeria: Its Petroleum Geology, Resources and Potential. Graham and Trotman, London



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