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Characterization of Local Alkali, Surfactant and Polymer Used For Enhanced Oil Recovery

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Abstract— **EDXRF analysis on akanwu, unpurified and purified palm bunch ash (Elaeis guineensis), saw dust ash (Khaya ivorensis), plantain peel ash (Musa sapientum), pawpaw leaves (Carica papaya), bitter leaf (Vernonia amygdalina), two species ogbonno (Irvingia gabonensis), achi (Brachystegia eurycoma), ofor (Detarium microcarpum), ukpo (Mucuna flagellipes) and okro (Abelmoschus esculentus) used as local agents for Enhanced Oil Recovery beyond conventional waterflooding showed the presence of major, minor and trace elements with its oxide in varying proportions. Unpurified palm bunch ash, purified palm bunch ash, saw dust ash, plantain peel ash and akanwu contain 56.03%, 56.996%, 22.41%, 54.15%, 12.65% and 3.55%, 4.91%, 0.512%, 2.64%, 5.877% of potassium oxide and sodium oxide respectively. The Loss On Ignition (LOI) test showed the presence of carbonate ions as 42.65%, 47.44%, 37.54%, 40.33% and 30.54% in unpurified palm bunch ash, purified palm bunch ash, saw dust ash, plantain peel ash and akanwu respectively. This analysis confirmed that the local akanwu in Nigeria is Potash and not Trona as commonly mistaken. pH is a major criteria for a successful alkaline flooding. Pawpaw leaves and bitter leaf contains 56.41%, 22.66%, 1.66%, 1.37% and 53.14%, 27.28%, 0.66%, 5.68% of potassium oxide, calcium oxide, iron III oxide and sodium oxide respectively. Aluminium and lead were absent in both unpurified and purified palm bunch ash but lead was present in plantain peel ash and akanwu sediment, though absent in extract from akanwu but silicon oxide, aluminium oxide and titanium oxide were present in akanwu. Analysis on the local polymers used as food thickeners also showed that they contain major elements like potassium, calcium, sulphur, phosphorus and sodium but the absence of magnesium except for one of the species of ogbonno. Pawpaw leaves contain vital elements and oxide that makes it nutritional rich for human consumption like other vegetables. These local materials if modified and refined can substitute synthetic chemicals used for Enhanced Oil Recovery.**

Index Terms— **local materials, major elements/oxides, minor elements/oxides, pH, Enhanced Oil Recovery, EDXRF**

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I. INTRODUCTION

As the demand for energy keeps increasing, it becomes imperative to scoop the reservoir, thereby producing trapped and mobile oil to meet the increasing demand for energy. Chemical flooding is an aspect of enhanced oil recovery whereby chemicals (Alkaline, Surfactant and Polymer) are introduced into the reservoir to release and produce trapped and mobile oil that remained after primary or secondary recovery due to viscous, gravity and capillary forces. The success of this process has contributed immensely to additional recovery. One of the demerits of this type of enhanced oil recovery method is the high cost of the chemicals which makes the process worthwhile only when there is hike on oil price. As the Niger Delta prepares for tertiary recovery stage, it becomes important to introduce local contents as substitutes for the high cost chemicals for sustainability of the process. Research has shown that some local materials in Nigeria contain chemical compounds that can serve as Alkaline, Surfactant and Polymer when modified or refined. Since these local materials are renewable and cheap, interest in their potential abilities will make their sustenance more achievable.

II. LITERATURE REVIEW

Enhanced Oil Recovery is the use of other techniques beyond the conventional methods to recover extra oil from the reservoir. It comprises of five distinctive methods namely miscible, thermal, microbial and chemical flooding processes. The miscible flooding process is further divided into single and multiple contact process. In single contact process, alcohol or inert gases are used to achieve immediate miscibility for Enhanced Oil Recovery while in multiple contact process; methane is employed as it's immiscible at first contact but relies on chemical exchange between phases to achieve miscibility for Enhanced Oil Recovery. The thermal flooding process is the use of hot water flooding, cyclic steam injection and insitu combustion to increase the temperature in the reservoir for easy flow of oil. Microbial flooding process is the use of inherent or injected microbes to form polymer or surfactant in the reservoir, thus, enhancing oil recovery. Microbial flooding is yet to make commercial impact (Saleem *et al.,* 2011). Figure 1 shows the chemical flooding enhanced oil recovery process which is the area of interest in this research. It employs the characteristics of Alkaline, Surfactant, Polymer and its various blends to enhance oil recovery. In summary, primary recovery recovers less than 30%, while secondary recovery recovers about 30 to 50% but tertiary recovery (EOR) techniques can recover greater than 50%, even up to 80% depending on the crude oil and reservoir type (Sunnil *et al.,* 2010).

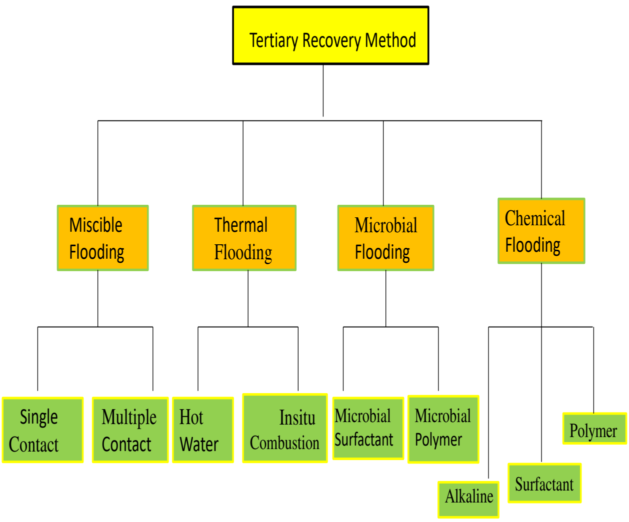

Figure 1: A schematic representation of Tertiary recovery methods

Since energy is as important as life itself and the demand keeps increasing with the growing population and human activities, it becomes imperative that we scoop the reservoir to release trapped and mobile oil that remained in the reservoir after conventional recovery method. The oil displacement mechanisms through which chemical flooding enhance oil recovery includes reduction in interfacial tension between formation water and oil, increase in viscosity of displaced fluid, reduction in capillary pressure, improved sweep efficiency, wettability alteration of the formation rock, reduction in mobility ratio between displacing and displaced fluid, emulsification of oil, pH, salinity control and permeability alteration. Like any other process, chemical flooding has its demerits. One of which includes high cost of the synthetic chemicals, hence, making the process worthwhile only when there is hike in oil price. Logistics involved in supply of these chemicals as at when needed is another determinant factor to its sustenance.

Research had shown that some local materials has the attributes of these synthetic chemicals and contain some chemical components used as alkali, surfactant and polymer which can be modified and refined. Since these local materials are renewable and cheap, interest in its potential abilities will make its sustenance more achievable. Local alkalis are materials that contain oxides or carbonate ions of sodium and/or potassium. These materials when dissolved in water form its hydroxide or carbonate ions. Such local materials are ash of palm bunch, plantain peel, coco pod, saw dust and *akanwu*. These local materials have the tendency of reacting with the acidic constituents of crude, majorly the organic acids in the crude to form an insitu-soap, which reduces the interfacial tension between oil and formation water. Local surfactants or co-solvents are materials with foaming and surface active effects, which further decrease the interfacial tension between formation water and oil to an ultra-low level. These local surfactants/co-solvents are pawpaw leaves extract, palm wine, local gin (kai-kai), bitter leaf, soybeans (lecithin) and jatropha seed. Local material polymers are mostly thickeners or viscosifiers that increase the viscosity of a fluid. Most of them are used as soup thickeners in Nigeria. These include ogbonno, achi, ofor, okobo, akparata, cocoyam, starch, ukpo and okro. These local materials when dissolved in water increases its viscosity. A research on ukpo, achi and ofor showed the presence of major and trace elements in these food thickeners. They contain elements like Potassium, Calcium, Manganese, Iron, Nickel, Copper, Zinc and Sodium (Onudibia *et al.,* 2014).

This research characterised these local materials found in Nigeria, West Africa (unpurified palm bunch ash, purified palm ash, plantain peel ash, saw dust ash, *akanwu*, *akanwu* sediment, paw-paw leaves, bitter leaf, achi, offor, ogbonno (two species), ukpo and okro) in order to determine the chemical components that are responsible for their efficiency as Enhanced Oil Recovery agents. Advance in this research will lead to a replacement of synthetic chemicals with local materials which will lead to increase in profit and a better life for the populous who will take it as an opportunity to invest in its cultivation.

III. METHODOLOGY

Palm bunch, unripe plantain peel and saw dust were burnt to ash while paw-paw leaves, bitter leaf, achi, ofor, ukpo, ogbonno 1 (from Ugiri seed), ogbonno 2 (M) and okro were dried and pulverished. Palm bunch ash was purified with a native leave which turned its colour from black to white. *Akanwu*; a naturally occurring mineral which is often called Trona /Potash/Natron was grinded to powdery form before use.

EXPERIMENTAL APPARATUS

- Porcelain crucible, furnace, desiccator, sample cup, sample changer, Energy Dispersive X-ray Fluorescence Spectrometer.

STEP-WISE PROCEDURE FOR CHARACTERIZATION OF LOCAL MATERIAL USING EDXRF SPECTROMETER

- The samples were prepared into a dried powdery form (even the pawpaw leaves were dried for weeks and pulverized; same goes to all the local materials used for this experiment)
- The dried powdery samples went through ashing under a controlled condition of temperature in a furnace in order to remove all the organic matters (carbon content)
- The powdered samples were weighed into a clean porcelain crucible. The quantity of the sample was calculated and placed in the furnace
- The furnace was ignited at 550°C for one hour thirty minutes (1hr 30mins)
- The ash content was then placed in a desiccator in order to cool down
- The ash content was carefully transferred into a sample cup and the sample cup with the sample were placed on a sample changer in a known position
- In the computer software for the EDXRF, click measure on the measure icon and select the position at which the sample was placed in order to bring the position on the focus as a target to be measured
- It took one hundred seconds (100secs) to analyse one sample

EXPERIMENTAL PROCEDURE FOR LOSS ON IGNITION (LOI) USING GRAVIMETRIC METHOD FOR DETERMINING THE PRESENCE OF CARBONATE ION

The main purpose of this experiment was to ascertain that a carbonate ion was actually decomposed into its oxide form; evolving carbon dioxide as a gas. The equation is as shown below:

$$K_2CO_3 \longrightarrow K_2O + CO_2\uparrow$$

$$Na_2CO_3 \longrightarrow Na_2O + CO_2\uparrow$$

- One gram (1g) of the sample was weighed into a clean weighed crucible
- The crucible and its sample was placed in a furnace and ignited at 1000°C for thirty to forty-five minutes (30-45mins)
- The crucible and sample were brought out and placed in a desiccator to cool down and then weighed
- The weight of the crucible plus sample before ignition was taken as A while the weight of the crucible plus sample after ignition was taken as B
- The Loss on Ignition was calculated as :

$$LOI = \frac{(A-B)g}{1g} \times 100\%$$

Note: 1g stated in the equation above is the one gram of the weighed sample in procedure one

RESULTS AND DISCUSSION

Table 1: MAJOR ELEMENTS AND ITS OXIDE IN THE LOCAL MATERIALS

SAMPLE ID	LOCAL MATERIAL	P %	K %	S %	Ca %	Mg %	Na %	P_2O_5 %	K_2O %	SO_3 %	CaO %	MgO %	Na_2O %
S1	PBA (UNPURIFIED)	1.95	46.50	1.26	11.60	N/D	2.64	4.47	56.03	3.15	16.22	N/D	3.55
S2	UKPO	7.62	41.80	1.82	9.56	N/D	0.74	17.48	50.37	4.55	13.37	N/D	0.997
S3	SDA	1.60	18.60	1.26	46.33	N/D	0.38	3.67	22.41	3.15	64.82	N/D	0.512
S4	PBA (PURIFIED)	1.29	47.30	0.787	11.6	N/D	3.64	2.96	56.996	1.97	16.23	N/D	4.91
S5	ACHI	4.00	10.40	<0.001	50.20	N/D	0.23	9.176	12.53	<0.001	70.23	N/D	0.31
S6	OGBONNO U.	4.50	37.30	4.01	23.20	N/D	2.32	10.323	44.95	10.03	32.46	N/D	3.13
S7	OFOR	8.86	20.60	<0.001	30.20	N/D	0.26	20.32	24.82	<0.001	42.25	N/D	0.35
S8	OGBONNO M.	8.20	40.0	2.08	19.10	0.5	0.62	18.81	48.20	5.20	26.72	0.5	0.84
S9	PAW PAW LEAF	2.23	47.0	1.98	16.20	N/D	1.02	5.12	56.64	4.95	22.66	N/D	1.37
S10	PPA	1.1	44.94	0.52	4.94	N/D	1.96	2.52	54.15	1.30	6.91	N/D	2.64
S11	BITTERLEAF	1.86	44.1	0.948	19.5	N/D	4.21	4.27	53.14	2.37	27.28	N/D	5.68
S12	OKRO	2.57	48.50	1.30	13.5	N/D	3.24	5.895	54.44	3.25	18.89	N/D	4.38
S13	*AKANWU*	<0.001	10.50	0.46	15.90	N/D	4.36	<0.001	12.65	1.15	22.24	N/D	5.877
S14	*AKANWU* SEDIMENT	<0.001	6.80	0.06	25.5	N/D	2.32	<0.001	8.19	0.15	35.67	N/D	3.13

Table 2 : MINOR ELEMENTS AND ITS OXIDE IN THE LOCAL MATERIALS

SAMPLE ID	LOCAL MATERIAL	Fe %	Cu ppm	Zn ppm	Cl %	Fe_2O_3 %	CuO ppm	ZnO ppm
S1	PBA (UNPURIFIED)	2.04	0.017 ppm	0.25 ppm	4.41	2.92	0.021 ppm	0.311 ppm
S2	UKPO	11.10	0.244 ppm	0.389 ppm	1.67	15.88	0.305 ppm	0.484 ppm
S3	SDA	7.70	0.073 ppm	0.21 ppm	1.99	11.02	0.091 ppm	0.261 ppm
S4	PBA (PURIFIED)	2.34	0.13 ppm	0.24 ppm	8.22	3.35	0.163 ppm	0.299 ppm
S5	ACHI	7.87	0.30 ppm	0.21 ppm	<0.001	11.26	0.375 ppm	0.261 ppm
S6	OGBONNO U.	2.19	0.16 ppm	0.267 ppm	7.86	3.13	0.200 ppm	0.332 ppm
S7	OFOR	13.5	0.58 ppm	0.51 ppm	<0.001	19.32	0.73 ppm	0.635 ppm
S8	OGBONNO M.	1.73	0.225 ppm	0.411ppm	1.96	2.47	0.282 ppm	0.512 ppm
S9	PAW-PAW LEAF	1.16	0.075 ppm	0.11 ppm	3.83	1.66	0.094 ppm	0.11 ppm
S10	PPA	8.06	0.064	0.099 ppm	4.64	11.53	0.081 ppm	0.099 ppm
S11	BITTERLEAF	0.461	0.067 ppm	0.130 ppm	8.11	0.66	0.084 ppm	0.137 ppm
S12	OKRO	1.63	0.092 ppm	0.21 ppm	5.32	2.33	0.115 ppm	0.261 ppm
S13	*AKANWU*	7.80	0.27 ppm	<0.001 ppm	7.87	11.16	0.338 ppm	<0.001 ppm
S14	*AKANWU* SEDIMENT	8.30	0.14	<0.001 ppm	3.96	11.88	0.175ppm	<0.001 ppm

Table 3 : OTHER ELEMENTS AND ITS OXIDE IN THE LOCAL MATERIALS

SAMPLE ID	LOCAL MATERIAL	SiO_2 %	Si %	PbO ppm	Pb ppm	Al_2O_3 %	Al %	TiO_2 %	Ti %	L.O.I %
S1	PBA (UNPURIFIED)	3.20	1.494	ND	ND	ND	ND	0.48	0.29	42.65
S2	UKPO	4.24	1.98	0.300	0.28	2.00	1.06	0.16	0.096	N/A
S3	SDA	4.13	1.93	0.53	0.49	0.24	0.13	0.45	0.27	37.54
S4	PBA (PURIFIED)	3.90	1.82	ND	ND	ND	ND	0.44	0.26	47.44
S5	ACHI	ND	ND	0.097	0.900	ND	ND	0.60	0.36	N/A
S6	OGBONNO U.	1.01	0.472	ND	ND	ND	ND	0.80	0.48	N/A
S7	OFOR	1.40	0.65	ND	ND	0.08	0.042	ND	ND	N/A
S8	OGBONNO M.	2.88	1.34	ND	ND	1.00	ND	0.18	0.11	N/A
S9	PAW-PAW LEAF	2.15	1.00	0.006	0.0056	ND	ND	0.06	0.036	N/A
S10	PPA	0.80	0.374	0.009	0.0083	ND	ND	0.18	0.11	40.33
S11	BITTERLEAF	ND	ND	ND	ND	ND	ND	0.05	0.03	N/A
S12	OKRO	ND	ND	ND	ND	ND	ND	0.06	0.036	N/A
S13	*AKANWU*	5.45	2.54	ND	ND	3.21	1.70	0.14	0.084	30.54
S14	*AKANWU* SEDIMENT	7.61	3.55	0.008	0.007	2.98	1.57	0.17	0.102	33.43

These local materials were characterized using an Energy Dispersive X-ray Fluorescence. The EDXRF is a device used to give elemental/elemental oxides composition of matters irrespective of the class whether organic or inorganic. It has advantage over Atomic Absorption Spectrometer (AAS) in that it gives both metallic and non-metallic content of a sample with its oxides while AAS gives only the metallic content of a sample without its oxides. EDXRF was chosen for this analysis because all the local materials (except one) in question are amorphous materials while X-ray diffractor is used to study crystalline structures mostly inorganic. Table 1, 2 and 3 showed that these local materials contain vital elements.

From this analysis, the unpurified palm bunch ash and the purified palm bunch ash (purified with a native leaf to fade off its colour) showed that they contain different composition, for example, the sodium oxide, phosphorus oxide, sulphur oxide, potassium oxide, calcium oxide, iron oxide, copper oxide and zinc oxide are 3.55%, 4.47%, 3.15%, 56.03%, 16.22%, 2.92%, 0.021ppm, 0.311ppm and 4.91%, 2.96%, 1.97%, 56.996%, 16.23%, 3.35%, 0.163ppm, 0.299ppm respectively. Magnesium oxide was not detected in both unpurified and purified palm bunch ash. This explains why the pH of the purified palm bunch ash (11.9) was higher than that of the unpurified palm bunch ash (11.2) since the percentage of potassium oxide (56.03%) was also higher than that in unpurified palm bunch ash (56.996%). The analysis also showed the reason why the soap made from our local alkali (palm bunch ash and plantain peel ash) are often soft because they contain more of potassium oxide than sodium oxide. Potassium oxide and sodium oxide in unpurified palm bunch ash, purified palm bunch ash, plantain peel ash are 56.03% & 3.55%, 56.996% & 4.91%, 54.15%% & 2.64% respectively. This also confirmed the why the pH for palm bunch ash solution (11.2) was higher than that of plantain peel ash solution (9.9) as a result of higher percentage of potassium oxide which dissolves in water to form potassium hydroxide. These local alkalis can best substitute synthetic materials since they contain high metallic oxide and exhibits high pH needed for Enhanced Oil recovery and other industrial uses. An experiment was carryout on Loss On Ignition (LOI) in order to affirm if a carbonate ion decomposed. The result confirmed that a carbonate ion was decomposed from the unpurified palm bunch ash, purified bunch ash and plantain peel ash. The percentage loss on ignition for unpurified palm bunch ash, purified bunch ash and plantain peel ash was given as 42.65%, 47.44% and 40.33% respectively (Table 3); which shows that the palm bunch ash contain more carbonate ion than the plantain peel ash. Saw dust ash has lesser percentage of potassium oxide (22.41%) and sodium oxide (0.512%); hence its low pH (9.5). Its loss on ignition (37.54%) was also low compared to that of palm bunch ash and plantain peel ash. Saw dust ash contains lead, silicon, aluminium and titanium among others (Table 3) in varying proportion.

Akanwu is a local Nigerian name used for Trona ($Na_2CO_3.Na_2CO_3.2H_2O$), Natron ($Na_2CO_3.NaHCO_3.2H_2O$), Soda ash (Na_2CO_3), Crystal Potash ($K_2CO_3.2H_2O$) or anhydrous Pearl Ash (K_2CO_3). The right name for *akanwu* should be a function of its chemical composition since all forms of this crystalline salt exhibits almost same characteristics. So from this analysis, it can be concluded that *akanwu* (used for this analysis) suggested to be Trona/Natron/Soda ash is actually crystal potash. Also the density of *akanwu* used for this analysis was 1.038g/cm^3 and the analysis from the characterization showed that *akanwu* contains high percentage of potassium oxide (12.65%) than sodium oxide (5.877%). Looking at the formular for Trona, Natron and Soda ash; It can be concluded that *akanwu* in Nigeria is crystal potash. This crystal potash also contains silicon, aluminium and titanium and its oxides. Only the sediment from *akanwu* contains some percentage of lead and its oxide (0.007ppm & 0.008ppm respectively). This shows that the extract from *akanwu* used mostly for Nigerian delicacy does not contain lead; so using *akanwu* fully should be discourage because its poison has to do with its dosage. The presence of iron oxide and aluminium oxide in higher quantity in *akanwu* confers the traditional Igbo practise during cooking that some natives throw in clean nail iron metals into time taken food stuffs during cooking to soften them easily and cook them faster. This is because iron oxide and aluminium oxide are used as catalyst to hasten a chemical reaction. It means that those spoons and nails emit iron oxide with the water which acts as a catalyst to quicken the cooking process. Surprisingly, *akanwu* which seems to have lesser percentage of potassium oxide than other local alkalis performed better than others during the Sand-pack flooding experiment. This could be attributed to the pH advantage of *akanwu*, since *akanwu* had a higher pH value (11.9) than palm bunch ash (11.2), plantain peel ash (9.9) and saw dust ash (9.5); meanwhile high pH value is the major criteria for a successful alkaline flooding.

The analysis showed that paw-paw leaves contain more potassium (56.64%) than bitterleaf (53.14%), no wonder its pH (9.8) was also higher than that of bitterleaf (5.4) which also made its oil recovery ability more effective than that of bitterleaf. In the early days, it was said that paw-paw leaves are used to soften meat; this analysis explains better that pawpaw leaves contain high percentage of potassium oxide that hydrolysis to potassium hydroxide which is an alkali.

The local material polymers too had high percentage of potassium oxide in the sequence of their performance during Sand-pack flooding experiment. Okro, ukpo, ogbonnoM, ogbonnoU, achi, ofor had 54.44%, 50.37%, 48.20%, 44.95%, 12.53%, 24.82% potassium oxide respectively. This analysis showed clearly that the two species of ogbonno analysed were not same in chemical composition (Table 1). Achi performed better than ofor because of the high swelling index of ofor which makes it swell in water causing plugging of the pores during flooding. Its sodium oxide percentage is also in the order of its performance.

OgbonnoM contains magnesium and its oxide (0.5%) while this element was not detected in ogbonnoU. The presence of magnesium oxide causes hardness of water. This could be the reason why ogbonnoM ceased when used at higher concentration and even its viscosity could not be measured using the Cannon-viscometer (150/601B).In this characterization, it was found that this local polymers that serve as soup thickeners do not contain lead and its oxide except for achi (0.097ppm & 0.900ppm) and ukpo (0.28ppm & 0.300ppm). Okro and bitterleaf do not contain heavy metals like silicon, lead and aluminium

CONCLUSION

From the analysis discussed above, the following conclusions were drawn:

- *Akanwu* in Nigeria is potash and not Trona as mistakenly called.
- Paw-paw leaves contain vital elements like other vegetables and can also be used soften meat in place of *akanwu*.
- Food thickeners in Nigeria can be modified and used for other industrial application other than for human consumption.
- These local materials contain chemical components that are responsible for their performance as Enhanced Oil Recovery agents.

NOMECLARTURE

PPA = Plantain Peel Ash
PBA = Palm Bunch Ash
SDA = Saw Dust Ash
UPBA = Unpurified Palm Bunch Ash
PPBA = Purified Palm Bunch Ash
LOI = Loss On Ignition
EOR = Enhanced Oil Recovery
AAS = Atomic Absorption Spectrometer
EDXRF= Energy Dispersive X-ray Fluorescence

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