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Upgrading Heavy Crude Oil Potentials through Microwave Assisted Distillation *(pp.137-147.)*

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Abstract: The use of microwave heating technique has shown tremendous research interest and potentials. As demand from oil is increasing worldwide, heavy crude oil are often priced at a discount to lighter ones due to low proportion of low- boiling constituents (light fractions) and high concentration of impurities which create many operational and quality problems. This work aimed to introduce a novel for improving low- boiling fractions yield using microwave irradiation without addition of chemicals or additives. Microwave oven model HR-7802D was employed and a laboratory atmospheric distillation was conducted on heavy crude oil after been irradiated with microwave for few minutes at various power rates. Distillation results showed that, microwave heating is very effective at low- power rates of 480 and 560Watts, which gave a higher products yield. Microwave heating is feasible, environmentally friendly and has the potential to be used as an alternative to conventional heating.
Key words: crude oil, distillation, microwave irradiation, distillates yield

1 INTRODUCTION

Crude oil serves as major source of energy in the world today as compared to other fuels such as coal and electricity. The most important use of crude oil is as source of fuels for domestic and industrial application. It has become the major raw material for the petrochemical industry for the manufacture of detergents, pharmaceuticals, fertilizers, synthetic fibres and many other products needed for human existence. It is also used for the manufacture of technical white oils, emulsifiers and insulating oils (Sabarathinam, 2005).

Crude oil is a mixture of a very large number of different hydrocarbons molecules, which are organic compounds of carbon and hydrogen atoms that may include from one to 60 carbon atoms, along with inorganic compound called heteroatom: sulphur, nitrogen, and trace metals such as iron, nickel, copper, vanadium etc (Erikh *et al.*, 1984; Ram, 2006). The properties of hydrocarbons depend on the number and arrangement of the carbon and hydrogen atoms in the molecules. The most commonly found molecules are alkanes (linear or branched), cycloalkanes, aromatic hydrocarbons, or more complicated chemicals like asphaltenes (OSHA, 1993). In petroleum systems, asphaltenes and resinous substances comprise a major portion of the interracially active component of the oil (Abdulrahman *et* *al.*, 2006a). Each petroleum variety has a unique mix of molecules, which define its physical and chemical properties, like colour and viscosity (OSHA, 1993). Crude oil differs from one another in a large number of chemical and physical properties, many of which play an important part in their refining and subsequent sale as refined products (Hanni, 2004).

Heavy crude are distinguish from lighter ones by their physical properties: higher viscosity, and specific gravity, as well as heavier molecular composition. It is dense and viscous due to the high presence of naphthenes and paraffines (Petroleum- *Wikipedia*, 2007). Heavy crude oil contains greater proportions of higher-boiling constituents such as lubricating oil (motor oils, lubricants, engine oil, cylindrical oil, and gear oil), greases and wax, and residue (residual fuel oils, coke, tar and asphalt). In addition, more aromatic, and heteroatom – containing (N, O, S and metals) are contained in heavy petroleum than light petroleum (Speight, 2001). Consequently, heavy crude are often priced at a discount to lighter ones, probably due to increased refining costs, low proportions of lower- boiling fractions, and high sulphur content.

The refining process rearranges hydrocarbon structures and bounding patterns into different hydrocarbon molecules and compounds. Therefore, in the refining process, the type of hydrocarbon (paraffinic, naphthenic, or aromatic) is significant rather than its specific chemical compounds. These hydrocarbon molecules are separated by fractional distillation at an oil refinery to produce gasoline (petrol), jet fuel, kerosene, and other hydrocarbons such as 2,2,4 – trimethyl pentane (isooctane) widely used in gasoline (OSHA, 1993).

1.1 Crude Oil Refining

Distillation is one of the process of crude oil refining is principally based upon the differences in the boiling range of group of hydrocarbon. More often now the process is that of separating the fractions by "fractional condensation" which basically consists of heating crude oil to certain controlled temperature; so that most of the components turn into vapours and then condensating them at designed points to form various fractions. This technique of separation by distillation was in use long time ago. Hence crude oil is a mixture of hydrocarbons that boils or vapourises at different temperature; heating it to a certain temperature allows one fraction to boil off while other remain liquid (Kate, 1997). However, separation processes account for 40- 70% of both the capital and operating costs of a broad range of industries; and distillation is the most widely used of all the separation operation accounting for more than 95% applications than all the others worldwide. Consequently, more than 95% of the energy consumed by separation processes in the chemical process industries goes for distillation. Although, separation processes such as: membrane- based routes, adsorption and crystallization are not energy intensive (Sabarathinam, 2005).

Today, the energy requirement of distillation process requires an urgent attention to find a suitable means of producing energy with low cost. Distillation process uses energy from fuel fired source which posed serious environmental effect by their combustion. However, fuel and energy costs are major factors of the direct costs of refining crude oil and as a results of large increases in crude oils and natural gas prices; there is a great incentive to conserve fuel by utilizing radiation energy from other source. The radiation energy from different source varies widely. The energy available from radiation is in orders of magnitude higher than the activation energy needed for breaking a carbon- carbon, carbon-hydrogen, carbon- sulphur or carbon- nitrogen bond.

Microwave heating has an incontestable place in analytical and organic laboratory practices as a very effective and non-polluting method of activation and heating. There are numerous examples of applications of this technology in sample digestion, organic synthesis, analytical chemistry, water treatment and the food industry (Phutdhawong *et al.*, 2006; Zhang *et al.*, 2006). Microwave energy, with a frequency of 2.45 GHz, is well known to have a significant effect on the rates of a variety of processes. The number of reported applications, especially in the food industry, and analytical chemistry is increasing rapidly (Zhang *et al.,* 2006). In addition, Exxon Research and Engineering of New Jersey has patented a process that turn uneconomic shale oil and tar sand, of which there are vast deposits in the world, into useful hydrocarbons and ethylene gas by inserting wires and bombarding it with microwaves. The microwaves cause the wires to discharge electricity, which converts the crude oil (tar) to high- grade fuels (John, 1993). As reported by Britten *et al.,* (2005), heavy petroleum upgrading has been on chemical, thermal and mechanical basis, which are very complex, expensive and generate by- products. Microwave irradiation has been used in the petroleum industry in the past, to inspect coiled tubing and line pipe. This study aimed at upgrading heavy crude oil to enhance its economic value and encourage refiners in its processing.

1.2 Microwave Heating Technology

Since 1969, industrial engineers began developing microwave heating techniques that avoid some limitations of conventional heating (Letellier *et al.*, 1999). Microwave heating is a quick and efficient method of heating materials that are difficult to heat by convection or infrared methods, so production rates increase and product quality improves. Microwave technology uses high frequency electromagnetic waves (300MGz to 300GHz), whose wavelength ranges from 1cm to 1m that pass through material and cause its molecules to oscillate, generating heat. Microwave heating, because of its volumetric heating effects, offers a faster processing rate. In conventional thermal processing, energy is transfer to the material through convection, conduction and radiation of heat from the surfaces of the material. In contrast, microwave energy is delivered directly to material through molecular interaction with the electromagnetic field (Abdulrahman *et al.*, 2006a).

In heat transfer, energy is transferred due to thermal gradients, but microwave heating is the transfer of electromagnetic energy to thermal energy and is energy conversion, rather than heat transfer (Thostenson and Chu, 1999). The energy transfer does not rely on diffusion of heat from the surfaces and it is possible to achieve rapid and uniform heating of thick materials. Abdulrahman *et al.* (2006b), described microwave energy as a non- ionizing radiation that causes molecular motion by migration of ions (ionic conduction) and dipole rotations.

1.2.1 Ionic conduction

This refers to the electrophoretic migration of ions under the influence of the changing electric field. The resistance offered by the solution to the migration of ions generates friction, which eventually heats up the solution (Vivekananda *et al.,* 2007). The conductivity principle is a much stronger effect than the dipolar rotation mechanism with regard to the heat- generating capacity. That's why the media containing ions are heated more efficiently by microwave than polar solvents (Kappe and Alexander, 2005). The effectiveness or rate of microwave heating of an ionic solution is a function of the concentration of ions in solution (Ruren *et al.,* 2007).

1.2.2 Dipole rotation (Interactions)

This simply means realignment of the dipoles of the molecule with the rapidly changing electric field. Heating is affected only at a frequency of 240 MHz. The electric component of the wave changes 4.9×10^4 times per second (Vivekananda *et al.,* 2007; Ruren *et al.,* 2007). Every time the solvent molecules tries to align itself with the electric field to keep itself in the same phase, but with the electrical component of the wave changing at such a rapid speed, the molecules fails to realign itself and starts vibrating which generates heat through frictional force. With frequency greater than 2450 MHz the electrical component even changes at a much higher speed as a result the molecules does not get sufficient time to even start to align itself with the external field as a result no heating occurs. If the frequency is less than 2450 MHz the electrical component changes at a much lower speed and the molecules get sufficient time to align itself with the electric field, thus there occurs no heating. The above mechanisms clearly indicate that only dielectric materials or solvents with permanent dipoles get heated up under microwave. The efficiency with which different solvents heat up under microwave depends on the dissipation factor, which is indeed the measure of the ability of the solvent to absorb microwave energy and pass it on as heat to the surrounding molecules. The dissipation factor is given by the equation:

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \qquad (1)$$

Where ε'' is the dielectric loss which indicates the efficiency of converting microwave energy to heat and ε' is the dielectric constant which is the measure of the ability to absorb microwave energy (Vivekananda *et al.,* 2007).

1.3 Microwave Irradiation

Microwave irradiation is attracting increasing attention as a tool for facilitating chemical reactions. It is often possible shorten reaction times dramatically as well as improve product conversion. With microwave irradiation, since the energy is interacting with the molecules at a very fast rate, the molecules do not have time to relax and the heat generated can be, for short times, much greater than the overall recorded temperature of the bulk reaction mixture. Britten *et al.* (2005) observed that this radiation causes induced reactions within the compounds, which results in chain scission and recombination. In essence, there will be sites of instantaneous localized superheating where reactions will take place much faster than in the bulk. This explains the rate acceleration seen when performing reactions under microwave irradiation (Nicholas and Rashid, 2008).

2 METHODOLOGY

2.1 Materials and Equipment

Some of the materials used include heavy crude oil, and distilled water. Among the equipments used include domestic microwave oven, thermochiller circulator, distillation flask, thermometer, condenser, measuring cylinder, heating mantle electrothermal, conical flask, and plastic corks.

2.2 Sample Collection

The Escravos Heavy crude oil used for this study was obtained from Kaduna Refinery and Petrochemical Company, Kachia Road, Nigerian National Petroleum Corporation (NNPC), Kaduna, Nigeria. The heavy crude oil was stored in a clean 10 liters plastic container and kept in the laboratory until required for use.

2.3 Experimental Procedure

300g of heavy crude oil sample was weighed into a 1000ml round bottom flask and placed in the modified domestic microwave oven. The microwave power was adjusted to 480Watts. The timer was turned first to its maximum (60 minutes) then back to the desired microwave irradiation time 5 minutes. The operation began as soon as the oven door was firmly closed to activate the sample energy. After the set time elapsed, microwave irradiated crude oil sample was immediately poured into a three neck 1000ml distillation flask placed on a heating mantle (Electrothermal Model MS9505) which was adequately lagged with metal cottons to minimize heat loss. The heating mantle was set to 90^{o}C and heat supplied to the system (set- up) gradually by the heater to raise the sample temperature. As soon as the first drop of the distillate was observed, the thermometer reading was recorded as the initial boiling point (IBP) and also the flask temperature was recorded. The heating was continued gradually until the temperature of the vapour reached 170^{o}C. The cut between the IBP and 170^{o}C was collected in a plastic sample bottle, weighed and refrigerated. The process was allowed to continue and fractional cuts of (170-240)^{o}C and (240- 350)^{o}C were collected, weighed and refrigerated respectively. The procedure was repeated at various microwave power rates: 560, 640 and 720Watts respectively. The whole procedure was repeated again for 7 and 9 minutes microwave irradiation times.

3 RESULTS AND DISCUSSIONS

3.1 Experimental Results

Table 1: Results of Atmospheric Distillation of Heavy Crude Oil Microwave Irradiated for 5 Minutes.

Fractional Cuts ^{o}C (Products)	**Conventional Heating Yield (wt %)**	**Microwave Radiation Power, Watts**							
		480		**560**		**640**		**720**	
		Yield (wt %)	**+/-**	**Yield (wt %)**	**+/-**	**Yield (wt %)**	**+/-**	**Yield (wt %)**	**+/-**
Gasoline (IBP-170)	0.36	0.48	+0.12	0.31	-0.05	0.25	-0.11	0.37	+0.01
Kerosene (170-240)	6.49	9.78	+3.29	7.26	+0.77	5.28	-1.21	5.02	-1.47
Diesel (240-350)	15.55	15.81	+0.26	15.78	+0.23	9.70	-5.85	10.53	-5.02
Sub Total	22.40	26.07	+3.67	23.35	+0.95	15.23	-7.17	15.92	-6.48
Residue (> 350)	75.43	72.00	-3.43	74.86	-0.57	83.43	+8.00	82.71	+7.28
Loss	2.16	1.93	-0.23	1.79	-0.37	1.34	-0.82	1.25	-0.91
Sub Total	77.59	73.93	-3.66	76.65	-0.94	84.77	+7.18	83.96	+6.37
Grand Total	99.99	100.00	0.00	100.00	0.00	100.00	0.00	99.88	0.00

Table 2: Results of Atmospheric Distillation of Heavy Crude Oil Microwave Irradiated for 7 Minutes.

Fractional Cuts ^{o}C (Products)	**Conventional Heating Yield (wt %)**	**Microwave Radiation Power, Watts**							
		480		**560**		**640**		**720**	
		Yield (wt %)	**+/-**	**Yield (wt**	**+/-**	**Yield (wt**	**+/-**	**Yield (wt %)**	**+/-**

				%)		%)			
Gasoline (IBP-170)	0.36	1.28	+0.92	0.39	+0.03	0.31	-0.05	0.20	-0.16
Kerosene (170-240)	6.49	8.69	+2.20	7.68	+1.19	5.28	-1.21	5.51	-0.98
Diesel (240-350)	15.55	15.62	+0.07	15.12	-0.43	9.76	-5.79	9.53	-6.02
Sub Total	22.40	25.59	+3.19	23.19	+0.79	15.35	-7.05	15.24	-7.16
Residue (> 350)	75.43	70.57	-4.86	74.86	-0.57	83.14	+7.71	83.14	+7.71
Loss	2.16	3.84	+1.68	1.94	-0.22	1.50	-0.66	1.62	-0.54
Sub Total	77.59	74.41	-3.18	76.80	-0.79	84.64	+7.05	84.76	+7.17
Grand Total	99.99	100.00	0.00	99.99	0.00	99.99	0.00	100.00	0.00

Table 3: Results of Atmospheric Distillation of Heavy Crude Oil Microwave Irradiated for 9 Minutes.

Fractional Cuts ^{o}C (Products)	**Conventional Heating Yield (wt %)**	**Microwave Radiation Power, Watts**							
		480		**560**		**640**		**720**	
		Yield (wt %)	**+/-**	**Yield (wt %)**	**+/-**	**Yield (wt %)**	**+/-**	**Yield (wt %)**	**+/-**
Gasoline (IBP-170)	0.36	0.19	+0.17	0.59	+023	0.33	-0.03	0.31	-0.05
Kerosene (170-240)	6.49	9.25	+2.76	7.75	+1.26	5.62	-0.87	5.92	-0.57
Diesel (240-350)	15.55	16.92	+1.37	16.57	+1.02	10.42	-5.13	11.02	-4.53
Sub Total	22.40	26.36	+3.96	24.91	+2.51	16.37	-6.03	17.25	-5.15

Residue (> 350)	75.43	72.00	-3.43	73.14	-2.29	82.43	+7.00	81.28	+5.85
Loss	2.16	1.63	-0.53	1.95	-0.21	1.19	-0.97	1.46	-0.70
Sub Total	77.59	73.63	-3.96	75.09	-2.50	83.62	+6.03	82.74	+5.15
Grand Total	99.99	100.00	0.00	100.00	0.00	99.99	0.00	99.99	0.00

3.2 Discussion of Results

Table 1 summarizes the results of the atmospheric distillation of heavy crude oil microwave irradiated at various power rates for 5 minutes. The light distillate yields obtained are 26.07, 23.35, 15.23 and 15.92wt% at 480, 560, 640 and 720W respectively. At lower power rates (480 and 560W), the distillate yields increased by 3.67 and 0.95wt% as compared to 22.40wt% of distillate yields obtained by conventional heating (non- irradiated); and this left residues 72.00 and 74.86wt% at 480 and 560W respectively compared to 75.43wt% obtained under conventional heating (non- irradiated) operations.

In contrast, at high power rates (640 and 720W) the distillate yields decreased greatly by 7.17 and 6.48wt%; this caused residues to increase by 8.00 and 7.28wt% as compared to residue under conventional heating (non- irradiated). It is observed that, the distillate yields decreases as power rates increases; the reason for the observed decrease in the yield is not entirely clear but, could be attributed to a literature reported by Eyong *et al.* (2004) that 'newer models of microwave ovens have inverter power supply which used pulse width modulation that truly provide continuous low power microwave heating'. Probably, that was why at low power rates there are high distillate yields due to continuous microwave radiation, which causes scission of the high molecular weight hydrocarbon chains into smaller fractions that seem to recombine and form larger, longer, branched carbon chains as reported by Britten *et al.* (2005); and results to high yields. In support of the observation, a report by Abdulrahman *et al.*, (2006b) that 'the dielectric and loss factor of crude oil are not constant because they vary with temperature, frequency (power), and moisture content. The dielectric of a materials or combination of materials affects how the transmitted microwaves react with the materials in the system'. This may explain why at high power rates the dielectric properties of the crude oil vary hence not constant and consequently, affect the transmitted microwaves reaction with the crude oil material resulting in low fractions yield.

Table 2 presents the results of the atmospheric distillation of heavy crude oil microwave irradiated for 7 minutes at various power rates. The total percent distillate yields of 25.59, 23.19, 15.35 and 15.24wt% were obtained when irradiated at 480, 560, 640 and 720W, followed similar trend to results presented in Table 1. As can be observed, an increment of 3.19 and 0.79 wt% distillate yields was achieved still at low power rates (480 and 560W) as compared to 22.40wt% distillate yield under conventional heating (non- irradiated).

Whereas, distillate yields observed at high power rates (560 and 720W) decreased by 7.05 and 7.16wt% respectively. In the present Table, distillates yields observed were obviously lower compared to when irradiated for 5 minutes. On residues left, despite each distillate yields were determined independently, the same 83.14wt% residues were obtained at high power rates (640 and 720W); about 7.71wt% increment over 75.43wt% residue obtained by conventional heating (non- irradiated).

In contrast, at low power rate, the residues 70.57 and 74.86wt%, decreased by 4.86 and 0.57wt% respectively as compared to residue observed under conventional heating operation. A literature reported by Eyong *et al.* (2004) that ‘In newer microwave oven models (domestic type), magnetron (a device which generates microwaves) is turned on and off in cycles of several seconds at a time when operated at high power rate there is change in the intensity of radiation’. This explains why high power rates radiation might not facilitate hydrocarbon fragmentation (scission) caused by radiation as fast compared to low power rates. In support of the view, Vivekananda *et al.* (2007) in their literature found that varying microwave power from 600 to 1200 W had no significant effects on the variety of processes.

Table 3 summarizes the results for atmospheric distillation of heavy crude oil microwave irradiated for 9 minutes. The distillate yields obtained are 26.36, 24.91, 16.37 and 17.25wt% when irradiated at microwave power rates 480, 560, 640 and 720W respectively. These results show the same pattern as in Table 1 and 2 with a remarkable increase in distillate yields by 3.96 and 2.51wt% at low power rates (480 and 560W) was achieved compared to distillate yields when irradiated for 5 minutes. In similar contrast, at high power rates (640 and 720W) distillate yields decrease by 6.03 and 5.15wt% compared to low power rates. A literature reported by Vivekananda *et al.* (2007) that ‘microwave power and irradiation time are two factors, which influence each other to a great extent’. It is obvious, distillates yields were enhanced when irradiated at low power rates (480 and 560 W) for 5 and 9 minutes.

4 CONCLUSION

Results of this study show that, microwave radiation can be an effective tool to improve light distillate yields of atmospheric distillation without addition of chemicals or additives hence it affects hydrocarbon chains. However, continuous and effective heating can be achieved at low- power rates between 480 to 560Watts. Microwave heating at 480Watt for 9 minutes irradiation time gave a better distillate yield of 3.96wt% over conventional heating. As observed, distillates yield decreases with increase in microwave power level to 720W. These distillates yields result support Britten *et al.* (2005) conclusion that microwave radiation had caused fragmentation (scission) of higher molecular weight carbon chains and formation of free radicals (alkyls) which seem to recombined during zero energy activation (short relaxation time), forming smaller normal and branched carbon chains. This innovative microwave heating technique is feasible and has the potential to be used as an alternative to conventional heating which uses radiant heat from fuel- fired process in the furnace.

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