

EXPERIMENTAL INVESTIGATION OF FOULING CHARACTERISTICS OF DIESEL OIL

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ABSTRACT

This study investigated the fouling characteristics of diesel oil using hot-wire test equipment. The diesel oil was electrically preheated to a desired fluid temperature using a heating coil. The fluid entered the test section and swept through a nichrome wire which was supplied with the electrical current corresponding to the desired wire temperature. Fluid temperature of 46°C for wire temperatures of 200°C, 250°C and 300°C as well as fluid temperature of 63°C for wire temperatures of 250°C and 200°C were the operating conditions for the investigation. The heat transfer coefficients between the diesel oil and the heating coil were used to obtain the fouling resistance as a function of time. Fouling rates were later estimated for all the operating conditions. The result obtained showed that diesel oil fouled heavily and this fouling decreased with increasing fluid and wire temperature. Fouling was very severe for wire temperature of 200°C and fluid temperature of 46°C. The main mechanism of fouling of diesel oil was chemical reaction-polymerisation super-imposed with other fouling mechanisms.

Keywords: Fouling, Diesel Oil, Hot-wire Test.

INTRODUCTION

The ever-increasing energy cost has led to the pursuit of heat integration approaches in process industries in order to recover heat from the product streams as much as possible and thereby improve the energy efficiency of the plant. Generally, shell and tube heat exchangers are used as the heat recovery units. However, owing to the complex nature of crude oils, the heat exchangers in the crude preheat train in refineries are prone to fouling (Deshannavar *et al.*, 2010). The occurrence of fouling not only reduces refinery efficiency, but also the effectiveness of catalysts (Speight, 2015).

The term fouling is defined as the deposition of unwanted material on heat transfer equipment, which results in an increase in thermal resistance to heat transfer and subsequent loss of equipment thermal efficiency (Kovo, 2012; Samimi, *et al.*, 2013; Speight, 2014). Asphaltenes precipitation is considered to be a major cause of crude unit fouling (Fuhr *et al.*, 1991; Kovo, 2012).

Problems caused by the build-up of fouling in a refinery preheat train include: operating difficulties, economic/financial penalties, increased environmental impact, health and safety hazards (Coletti *et al.*, 2015). The total financial

penalties associated with fouling may include the loss of thermal efficiency in heat transfer equipment, high fluid pressure drops, high costs of anti foulant additives, physical and/or chemical cleaning and loss of production due to unscheduled plant shutdown (Rafeen *et al.*, 2010).

In theory, fouling can be divided into six mechanisms, which include: crystallization fouling, particulate fouling, chemical reaction fouling, corrosion fouling, biological fouling and solidification fouling (Lei *et al.*, 2011; Harche *et al.*, 2014), but in practice, it is often the interaction between two or more types. Epstein (1983) and Deshannavar *et al.* (2010) explained that the above fouling mechanisms generally occur in a series of steps namely: Initiation or Delay Period, Transport Period, Deposition Period, Removal Period and Aging Period.

The most important factors influencing fouling are: surface temperature, flow velocity, bulk temperature, crude type and crude blending (Srinivasan and Watkinson, 2005). The effect of surface temperature on the fouling rate is generally expressed by an Arrhenius-type equation as shown in equation 1.

$$\frac{dR_f}{dt} = A \exp(-E/RT_s) \quad (1)$$

where $\frac{dR_f}{dt}$ is the rate of fouling, A is the proportionality factor, E is the activation energy, R is the universal gas constant, T_s is the surface temperature. Saleh *et al.* (2005) calculated the activation energy values for a light Australian crude oil at film temperature and also at surface temperature. The use of film temperature or surface temperature depends upon the location of the chemical reaction. If the chemical reaction occurs in the thermal boundary layer, film temperature is used (Asomaning, 1997). On the other hand, surface temperature is used if the chemical reaction occurs on the heat transfer surface (Asomaning *et al.*, 2000).

Saleh *et al.* (2005) studied the effect of mixing and blending crude oils at certain operating conditions with the intention of using the results to guide a fouling mitigation strategy. Blending of crudes can cause unstable mixes which precipitate species such as asphaltene and result in rapid fouling (Wilson and Polley, 2001). The crude oil incompatibility and the precipitation of asphaltene on blending of crude oils can cause significant fouling and coking in crude preheat train. Hence, the crude oil compatibility model and tests were developed to predict proportions and order of blending of oils that would avoid incompatibility (Wiehe *et al.*, 2001).

Wang *et al.* (2015) presented a review of the experimental measurement and prediction models of crude oil fouling rate in crude refinery preheat trains. This review covered fouling development mechanisms, applications and limitations. Deshannavar *et al.* (2010) explained the different fouling mechanisms that crude oil undergoes at different stages of preheating as well as their mitigation strategies. It further expounded the various fouling models available, their advantages and limitations.

Kovo (2012) developed mathematical model to predict the fouling of Nigeria oil equipment installations, the modelling equation formulated was based on the principle of fouling process being in unsteady state condition (occurrence of accumulation). Harche *et al.* (2014) studied fouling

deposit in the heat exchangers of Algiers refinery, the work was a follow-up on the fouling resistance in heat exchangers of tube-calender type in Algiers' refinery.

MATERIALS AND METHODS

Materials

A hot-wire test equipment was used for the experiment. It is a laboratory-scale equipment which employs a technique called the modified hot-wire technique. It consists essentially of electrical heating of a metal wire (resistance nichrome wire) while in contact with the feed (the crude product under investigation: diesel oil). It employs a co-current flow system, which aligns the electrically-heated coil in the same direction as the liquid flows which represents a conduit flow typical of industrial processes.

Basically, most fouling rate measuring devices in current use are similar, while the industrial scale instrument provides an actual process heat transfer surface and mechanism. The laboratory-scale presents a representative model. However the laboratory-scale instruments have an overriding advantage of flexibility compared with the industrial devices. Thus, flow rate, composition, oxygen concentration, fluid and surface temperatures can be easily manipulated.

The laboratory-scale “Modified Hot Wire Technique” is in two forms: the cross flow system and co-current flow system. In the cross flow system, the electrically heated nichrome resistance wire stretches across the entire liquid cross-sectional area. Here, the contact time with the liquid is so short that the entire fouling procedure of chemical reaction, polymerization, crystallization and sedimentation will need to be instantaneous before changes due to fouling can be observed. On the other hand, the theory undermined the theory of deposition–removal, as particles are being removed as soon as they are deposited by the strong directed liquid shear forces.

The co-current flow system adopted in this research aligns the electrically heated coil in same direction as the liquid flows. This to some extent closely represents conduit flow typical of industrial processes. In this type of flow, contact

time is significant and as such all the fouling mechanisms are allowed to take place. The “Co-current Modified Hot Wire” fouling rate measuring instrument designed and used in this study is capable of operating within fluid velocity of 8 f.p.s, fluid temperature of 150°C, wall temperature of 1000°C and system pressure of 2 atmospheres absolute.

The process parameters (i.e. flow rate, composition, oxygen concentration and fluid and surface temperature) are still alterable, even when the system is under operation. Oxygen concentration in this system was monitored by providing a nitrogen blanket of known oxygen content on the liquid in the reserve (storage) tank.

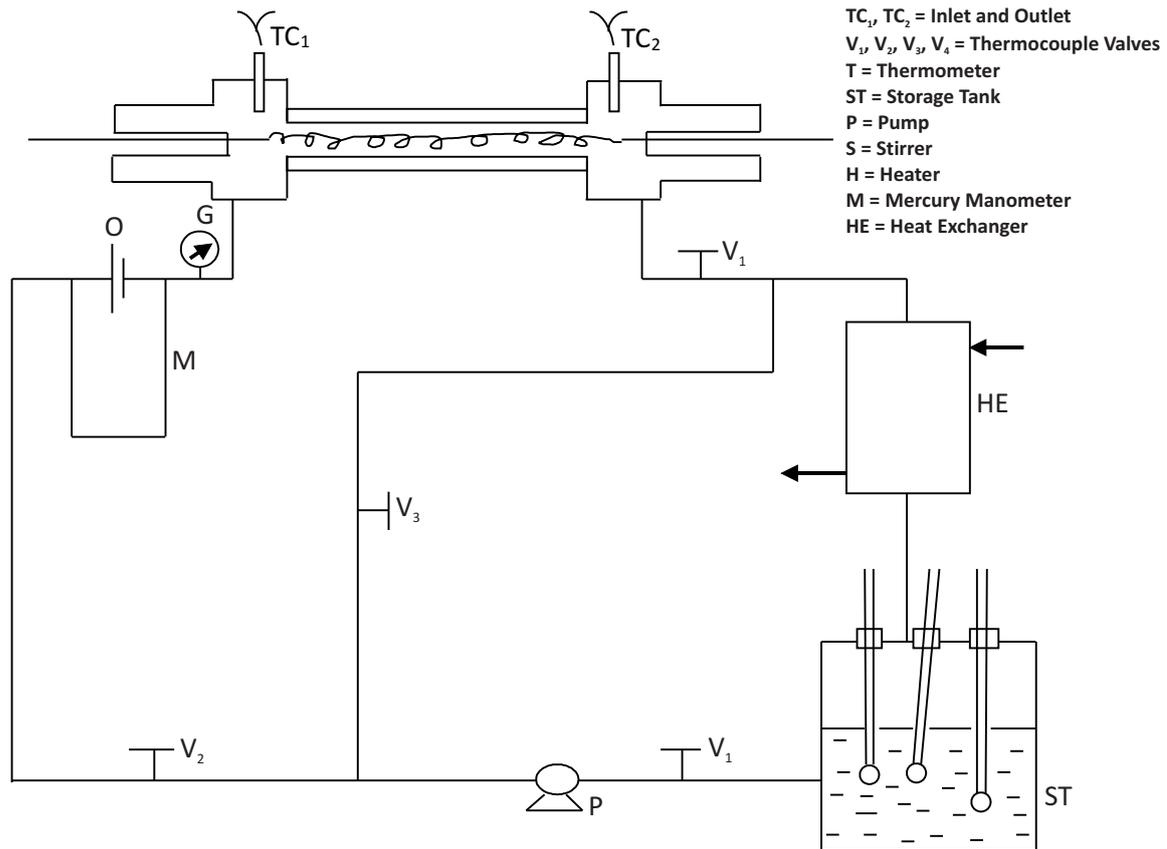


Figure 1: Schematic Diagram of Hot- Wire Test Equipment

The schematic diagram of the Hot-Wire Test Equipment is as shown in Figure 1. The materials used for the experiment which were the components of the equipment hot- wire test consist of the Test Section which is made from resistance nichrome wire, 18cm carbon steel tube (thermally insulated), 2 perforated “cm bolts and 2cm packing seal. It also consists of 2 thermocouples: copper – constantan and chromel – alumel as inlet and outlet thermocouples respectively. Ceramics rings were used as electrical shields that were placed at a contact point of pipe and nichrome wire. The flow meter is made up of mercury in tube manometer (Outside diameter OD, Inner diameter-ID = 2mm, U-length: 8cm

wide and length of 30cm) with orifice weir diameter: 0.25cm, and 10 atm F.S.O. pressure gauge: 0-1.0 kg/cm² (0.25 division). The Reserve Tank (storage tank) consist of Exchanger 2½ Gallon cylindrical tank (thermally insulated), a mercury in glass thermometer, 1000 watt electrical heating coil, 1.1 heat exchanger shell 9cm - OD, 8.25 ID, 60 cm length, tube no. 10 which is equal to 0.92 cm ID, 1.32 cm OD 55 cm length.

The Fluid Flow Materials consist of Centrifugal Pump (which has a volumetric rate of 11-54.5m³/s, an horse power of 840-150 HP), height of 10-45ft, and power rating of 220/240 volts, type- sartex pump). It is also made up of 4 globe

valves approximately 4m long, 1.32 cm OD, 0.92 ID carbon steel, and 1m long rubber hose pipes. The Electrical power supply system and measuring instruments consist of a variac of 230Volts 50 Hz, 5 Ampere fuse, 2 single throw switches, 1 Ammeter (0-10 Amp. AC, 50Hz), 1 Voltmeter (0-20) Volt AC, 50 Hz), and 1 Potentiometer 0.01 millivolt.

EXPERIMENTAL PROCEDURE

The experiment was carried out firstly by ensuring that all the bolts, elbows and seals were checked for any leaking points. The 2.5 gallon tank was filled with diesel oil heated up by an immersion heater. The fluid temperature in the tank was measured by mercury in glass thermometer. The fluid was pumped via a back-mix with valve V_2 and V_4 closed while V_1 and V_3 were opened. This was allowed to operate for 20 minutes while at the same time adjusting the cooling water valve to ensure that a constant temperature desired was achieved in the tank. When the temperature is fairly constant as indicated by the thermometer reading, valve V_3 was shut while valve V_2 and V_4 were opened. The variac switch was put on and adjusted to the current that gives the desired wire temperature.

The fluctuation in the wire resistance was basically due to movement inside the pipe. This corresponded to the power dissipated which was evaluated by measuring the wire resistance (R) using V/I . The Power was then estimated as I^2R .

The Heat dissipated by the nichrome wire, Q_M was then equal to I^2R . This equals the Heat transferred to the flowing diesel oil, Q_T as shown in equation

2.

$$Q_M = Q_T = mC_p\Delta T \quad (2)$$

Where, Q_M = Heat dissipated by the resistance nichrome wire (Cal/s), Q_T = Heat transferred to the flowing diesel oil (Cal/s), m = mass flowrate = volumetric flowrate \times Density of Diesel, ΔT = change in temperature ($^{\circ}C$).

The heat transfer coefficient, U was later evaluated in equation 3 as:

$$U = \frac{Q_T}{A\Delta T} \quad (3)$$

where, Heat transfer area, A = Circumference of wire \times Length of wire. Hence, heat transfer coefficient, U can be evaluated. The fouling resistance, R_f ($Cal/s.cm^2.^{\circ}C^{-1}$) is then evaluated in equation 4 as:

$$R_f = \frac{1}{U} - \frac{1}{U_o} \quad (4)$$

Where, U_o = initial (clean) heat transfer coefficient

RESULTS AND DISCUSSION

Table 1 shows the operating conditions (wire temperatures, T_w and fluid temperatures, T_f) for the five runs considered in the experiment. The first three runs have the same fluid temperature (T_f) of $46^{\circ}C$ but different wire temperatures (T_w) of $200^{\circ}C$, $250^{\circ}C$ and $300^{\circ}C$. The last two runs also have the same fluid temperature of $63^{\circ}C$ but different wire temperatures of $250^{\circ}C$ and $200^{\circ}C$. Table 2 shows the values of fouling resistance of each run as a function of time.

Table 1: Wire and Fluid Temperatures of each Run

Run	Wire Temperature, T_w ($^{\circ}C$)	Fluid Temperature, T_f ($^{\circ}C$)
1	200	46
2	250	46
3	300	46
4	250	63
5	200	63

Table 2: Fouling Resistance Data R_f versus Time

Time (h)	R_f (Cal/s.cm ² . °C) ⁻¹				
	Run 1	Run 2	Run 3	Run 4	Run 5
0	0	0	0	0	0
1	1.65	0	2.42	13.50	0
2	2.73	1.22	0	14.20	2.85
3	142.91	2.79	1.25	17.55	11.44
4	49.25	3.43	39.72	22.37	2.39
5	77.39	7.22	65.75	35.39	14.11
6	105.97	21.15	74.30	47.01	15.27
7	171.93	77.55	77.65	30.49	42.77
8	195.30	89.22	101.22	37.27	49.55
9	132.55	145.50	190.99	110.45	57.22
10	185.34	146.21	175.00	119.22	59.43
11	439.50	139.70	192.22	175.15	121.77
12	990.645	143.10	200.15	192.33	135.91
13	998.32	173.30	200.65	198.52	172.20
14	999.55	185.01	215.05	198.60	175.45

Table 3: Fouling Rates for each Run

Time (h)	Fouling Rate , $\Delta R_f / \Delta t$ (Cal/s ² .cm ² . °C) ⁻¹				
	Run 1 $T_w = 200^\circ\text{C},$ $T_f = 46^\circ\text{C}$	Run 2 $T_w = 250^\circ\text{C},$ $T_f = 46^\circ\text{C}$	Run 3 $T_w = 300^\circ\text{C},$ $T_f = 46^\circ\text{C}$	Run 4 $T_w = 250^\circ\text{C},$ $T_f = 63^\circ\text{C}$	Run 5 $T_w = 200^\circ\text{C},$ $T_f = 63^\circ\text{C}$
0 – 3	47.64	0.93	0.42	5.85	3.81
4 – 6	28.36	9.18	17.19	12.32	1.58
7	24.56	11.08	11.09	4.36	6.11
8	24.41	11.15	12.65	4.66	6.20
9	14.73	18.24	21.22	12.27	12.80
10	18.53	14.62	17.50	11.92	5.94
11-14	186.68	15.08	7.76	7.82	17.89

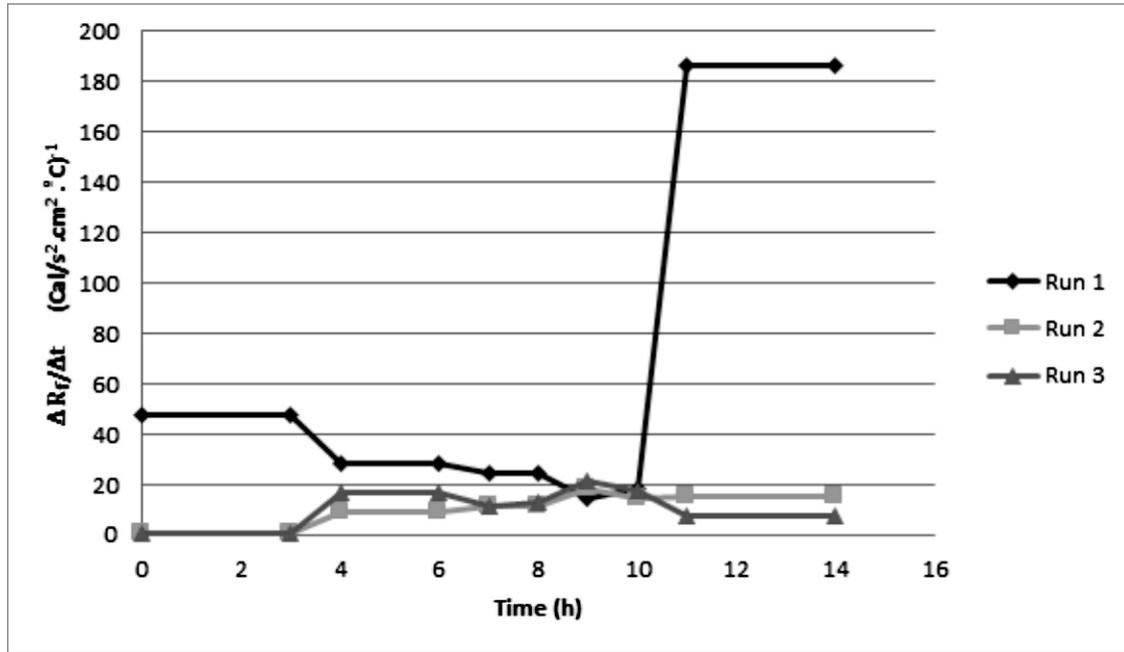


Figure 2: Graph of Fouling Rate ($\Delta R_f/\Delta t$ versus Time) for Run 1, 2 and 3

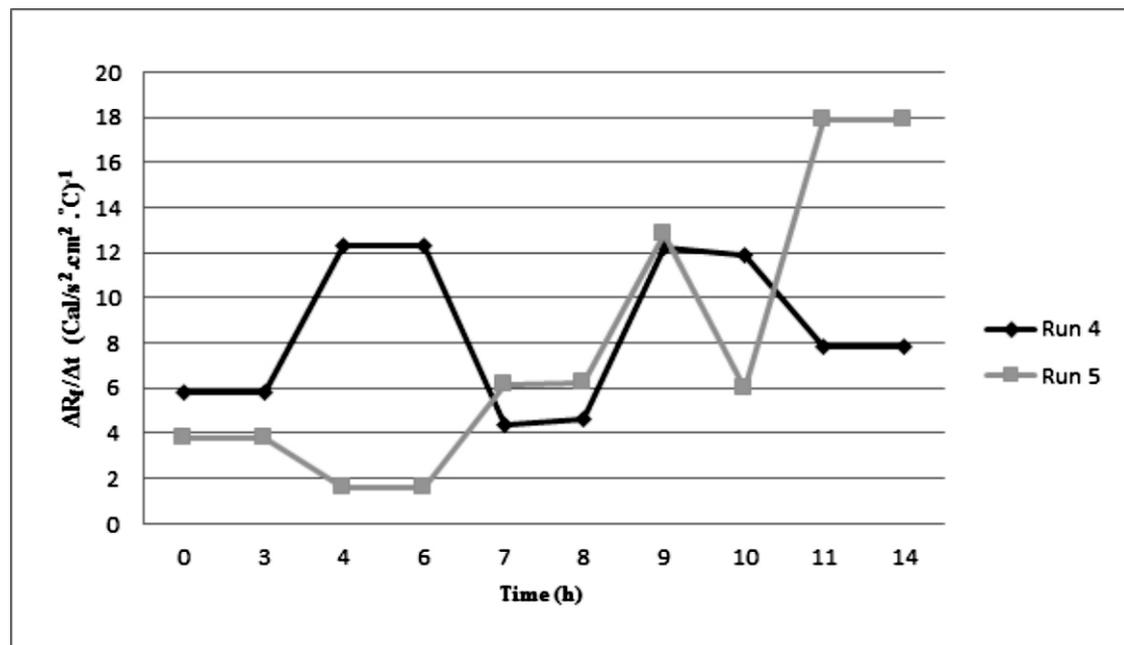


Figure 3: Graph of Fouling Rate ($\Delta R_f/\Delta t$ versus Time) for Run 4 and 5

Table 2 estimated the fouling resistance (R_f) while Table 3 estimated the fouling rate ($\Delta R_f/\Delta t$) which is fouling resistance per time ($\text{Cal/s}^2.\text{cm}^2.\text{°C}^{-1}$). Figures 2 and 3 were obtained from Table 3 having evaluated fouling rate ($\Delta R_f/\Delta t$).

The fouling rates ($\Delta R_f/\Delta t$) for all the five (5) runs were shown in figures 2 and 3. As shown in Table 3, the fouling rate ($\Delta R_f/\Delta t$) for run 1 was observed

to increase from 47.64 ($\text{Cal/s}^2.\text{cm}^2.\text{°C}^{-1}$) at starting point (0-3)h and decreased to 14.73 at 9h but finally reached 186.68 ($\text{Cal/s}^2.\text{cm}^2.\text{°C}^{-1}$) at 11-14h. For run 2, the fouling rate increased from 0.93 at (0-3) h and reached peak value of 15.08 ($\text{Cal/s}^2.\text{cm}^2.\text{°C}^{-1}$) at (11-14) h. For run 3, fouling rate reached highest value of 21.22 ($\text{Cal/s}^2.\text{cm}^2.\text{°C}^{-1}$) at 9h but decreased to 7.76 ($\text{Cal/s}^2.\text{cm}^2.\text{°C}^{-1}$) at (11-14) h. For run 4, fouling rate started with initial

value of $5.85 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at (0-3)h, reached highest value of $12.32 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at (4-6)h before it decreased to $7.82 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at (11-14)h. For run 5, there was progressive increase from $1.58 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at (4-6)h to $12.80 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at 9h before it decreased to $5.94 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at 10h and later increased again to $17.89 \text{ (Cal/s}^2\text{.cm}^2\text{.}^\circ\text{C)}^{-1}$ at (11-14)h. This showed that the values of rates of fouling are not uniform which is expected for chemical-polymerization of fouling of hydrocarbon stream.

During the experiment, it was clear that the golden-oil colour of diesel oil became brown at first before it finally turned deep brown. From Table 3, it was also observed that the fouling rates were more significant for the lowest wire temperature (run 1) compared with run 3 (highest wire temperature). This was not the same trend for fluid temperature of 63°C where fouling rate was higher for a wire temperature of 250°C compared with wire temperature of 200°C

The general equation form of the reaction rate-controlled deposition shows that the rate of fouling is inversely proportional to the surface temperature. The surface temperature in the experiment is the wire temperature. At fluid temperature of 46°C in Figure 2, the fouling rate at wire temperature of 200°C was the highest and the lowest fouling rate was at 300°C . This trend was not observed for fluid temperature of 63°C . However, it can be established that lower surface temperature increased fouling

CONCLUSION

The non-uniform trend of fouling rate of diesel oil showed that mechanism of fouling rate were superimposed on chemical-polymerization fouling. The fouling rate was highest at a lower wire temperature for a fluid temperature of 46°C , but slightly higher at higher wire temperature for a fluid temperature of 63°C . A change in colour of diesel oil from golden-oil to brownish colour revealed that chemical reaction took place during the experiment.

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