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ORIGINAL ARTICLE

Effect of hydrogenation temperature on the palm mid-fraction fatty acids composition and conversion

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Abstract Palm mid fraction (PMF) was hydrogenated using Armfield hydrogenation pilot plant unit. Effects of hydrogenation temperature on the total conversion and selectivity were investigated. Noticeable effect of hydrogenation temperature on the reaction conversion was found. The values of reaction conversion at temperatures 99 and 130 °C were found to be considerably lower, when compared to those at temperatures 137 and 149 °C. The alteration on fatty acid composition had been studied at varying temperature and other parameters were kept constant. It can also be observed that the trans-fatty acids (TFA) content in the PMF samples hydrogenated at temperature ranges 130–137 °C, and 137–149 °C were found to be 0.197 and 0.186, respectively. Effect of reaction temperature on mass transfer resistance from the hydrogen bubble to the catalyst surface was estimated. The obtained results clarify that the volumetric mass transfer coefficient was increased around 140% by increasing the reaction temperature from 99 to 130 °C.

1. Introduction

As one of the 17 major oils traded in the global edible oils and fats market, palm oil (PO) and palm kernel oil contributed the highest percentage (28%) of the total global production of oils and fats in 2006. They also were the most tradable types of oils in the global oil market with a market share of 56% in the same year (MPOB, 2007). Palm oil and its fractions now take second place in the list of oils production and will probably overtake soybean oil in another 10–15 years (Gunstone, 2002). Palm oil, as a major product during processing of oil palm fruit, is obtained from the mesocarp or pulp of the palm

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Nomenclature

F_i	the moles number of the unsaturated fatty acid available in the PMF sample before the hydrogenation process
F_o	the mole numbers of the unsaturated fatty acids available in the PMF sample after the hydrogenation process
X	conversion
k_s	mass transfer coefficient (m/min)
D_i	diffusivity of hydrogen in the PMF (m^2/s)
ρ_l	PMF density (kg/m^3)
ρ_{cat}	The apparent density of Ni catalyst = 2200 kg/m^3
d_p	Mean particle diameter of Ni = 3×10^{-5} m
g	Acceleration due to the gravity = 9.81 m/s^2
μ	PMF viscosity (N.slm^2)
C_{H2}	Hydrogen concentration (kmol/m^3)
r_{H2}	rate of reaction per volume of solution (k mol H_2/m^3 min)
k_b	mass transfer coefficient for hydrogen absorption from the H_2 bubble to PMF (m/min)
a_b	bubble surface area (m^2 / m^3 of solution)
m	mass concentration of Ni catalyst (kg Ni/m^3 of solution)
k_c	mass transfer coefficient for hydrogen absorption from PMF to the external surface of Ni catalyst (m/min)
a_c	external surface of Ni (m^2/kg catalyst)
k	specific reaction rate (m^3/kg$_{cat}$ min)
η	effectiveness factor

fruit. It has been consumed as a food for 5000 years and today is found in one out of every ten food products worldwide (MPOB, 2007). Production and exports are dominated by two South East Asian countries (Malaysia and Indonesia). Malaysia has 51% of all palm oil production and 63% of palm oil export; Indonesia corresponding to 31% and 26%, respectively (Gunstone, 2002). Palm oil is a balanced oil as it contains equal amounts of both unsaturated (mainly oleic acid, C18:1 – 39%) and saturated (mainly palmitic acid, C16:0 – 44%). This property makes palm oil suitable raw material for producing oil and fat fractions with different physical and thermal properties (Gunstone, 2002; Chong, 1994). These fractions can be two components, i.e., a solid (stearin) and liquid (olein) fractions. Palm oil and palm kernel oil can be fractionated for different food applications (MPOB, 2007). Palm mid fraction (PMF) has the most uses of palm oil in food and nonfood applications (Schmidt and Schomaker, 2007; Bernas et al., 2009). Only 18-carbon fatty acids (Linolenic – C18:3, Linoleic – C18:2 and Oleic – C18:1) are suitable for palm mid fraction hydrogenation (Xiao, 2007). After partial hydrogenation, PMF gets a melting behavior and oxidation stability makes it suitable as a bakery fat and biscuit manufacturing (MPOB, 2007). PMF has a very high content in symmetrical di-saturated triglycerides (mainly POP) which is the basis of virtually all true coca butter equivalent (CBE's) on the market and is also very suitable for use in chocolate covered centers in place of lauric fats, which pose the risk of soapy rancidity when used improperly (Gunstone, 2002). CBE is also suitable for various food industries especially chocolate and ice cream products (Farahany et al., 2008). The high nutritional value of PMF in term of tocopherol and tocotrienols has been added to the (CBEs) products.

Since the first part of the 20th century, hydrogenation has been used to expand the application of vegetable oil in foods (Jang et al., 2005). The hydrogenation of edible oils is an important process in the fat industry because of its wide applications to produce margarine, shortenings, and frying oils (Murzin and Simakova, 2008). Hydrogenation is a process which hydrogen is chemically reacted with the unsaturated double bonds present in fatty acids (Xiao, 2007). Hydrogenation process is usually carried out in a three-phase semi batch reactor where hydrogen gas is bubbled with pressure in hot vegetable fat in the presence of a catalyst (Piqueras et al., 2006).

Oils have been hydrogenated since the 1930s to prolong their shelf life stability (Xiao, 2007). The chemical, physical and sensory properties of the final product strongly depends on the number of residual double bond and on the contents of cis-trans isomers present in the mixture (Ramirez et al., 2004), which depend on various operating factors, including temperature, hydrogen pressure, catalyst and circulation rate/agitation (Schmidt and Schomaker, 2007). The effect of temperature on the hydrogenation was clearly observed. Hydrogenation, like other chemical reactions, is accelerated by increase the temperature (Fernandez et al., 2007). Hydrogenated oil is more stable and does not become rancid as quickly as unhydrogenated oil (Murzin and Simakova, 2008). It has a higher melting point, and it is often used in frying and pastries for this reason. The basic hydrogenation process of edible oils means that the liquid oils change into semi-solid substances, which have desired melting characteristics and partially hydrogenated oils with increased stability (Pintauro et al., 2005).

This work aims to study the influence of hydrogenation temperature on the reaction conversion and fatty acid composition as well as estimation of temperature effect on mass transfer resistance of hydrogen molecule from the hydrogen bubble to the catalyst surface.

2. Experimental

2.1. Materials

All samples of refined bleached deodorized palm medium fraction (PMF) used in this study were supplied by Yemen Company for Ghee and Soap industry (YCGSI). PMF is produced from double fractionation process for the palm oil supplied from Malaysia/Indonesia or by single fractionation for palm olein. The fractionation plant used is Belgium origin, supplied by Tirtiaux-S.A. Fractionnement–Fleurus. The properties of palm mid fraction are shown in Table 1.

High purity hydrogen gas filled in cylinder of 150 bar was purchased from Saudi Arabia market and used in the experiments. Saturated steam of around 10 bar, generated by electrical boiler at the food science and technology department,

Table 1 Initial properties of unhydrogenated palm mid fraction (PMF).

Parameter		Value
Fatty acid composition (k mol/batch) × 10^3	C18:0	2.43
	C18:1	20.92
	C18:2	5.13
Trans-fatty acid content (k mol/batch) × 10^3	C18:1	0.005
	C18:2	0.16
	TOTAL	0.165

Faculty of Agriculture, Sana'a University, was utilized. The compressed air of 7 bar for the purpose of pneumatic mixer of nickel catalyst supplied to the pilot plant from compressor, which was a part of those constituting the pilot plant hydrogenation unit at the food science and technology department, faculty of agriculture, Sana'a university, was used. Tab water of 3 bar pressure was used for vacuum generating and cooling the hydrogenated PMF at the end of each experiment.

2.2. Catalyst

Commercial nickel/silica (Ni/SiO_2) catalyst was supplied by Jonson Matthey Catalyst. This catalyst is nickel dispersed on silica support and its properties are presented in Table 2.

2.3. The experimental set up

PMF samples were subjected to hydrogenation using the pilot plant (Armfield) presented in the food since and technology department at the faculty of agriculture, Sana'a university (Fig. 1). Such unit has been designed to reproduce all characteristics of the industrial unit, using a batch size of 25.0 l maximum. The reaction was controlled to occur at specified temperatures, hydrogen pressure and in the presence of nickel-based catalyst.

When reaction takes place, different degrees of saturation of the oils were attained through varying the process parameters. For all experiments, the PMF was filtered to separate the catalyst from the fat before carrying out the required analysis.

Due to low vapor pressure of PMF at the reaction temperatures (lower than 0.01 bar at 150 °C) (Yuan et al., 2005), it has been considered that the hydrogen partial pressure is the absolute reaction pressure.

The fatty acid and trans-fatty acid compositions of the unhydrogenated and hydrogenated samples were determined using GC technique. A column of high polar stationary phase, SP – 2340, 60 × 0.25 mm i.d., 0.20 μm film (Superlco Inc.) was used. The analyses have been carried out at Malaysian Palm Oil Board (MPOB) through Advanced Oleochemical Technology Division (AOTD) laboratories.

Table 2 Characteristics of commercial Ni/SiO_2 catalyst.

Ni (% wt)	22
SiO_2 (% wt)	4
Surface specific area (m^2/g)	188
Pore volume (ml/g)	0.23
Main pore diameter (Å)	20
*d*p (m)	3×10^{-5}
ρ_{apparent} (kg/m^3)	2200
ρ_{bulk} (kg/m^3)	2600

Reaction conversion is taken as the ratio of number unsaturated moles which have been saturated during the experiment to the total moles of the unsaturated available in the feed (unhydrogenated PMF).

$$\text{The conversion}\quad (X) = \frac{F_i - F_o}{F_i} \times 100 \tag{1}$$

It has been considered that one mole of linoleic acid (C18:2) contains two carbon–carbon double bond and one mole of oleic acid (C18:1) contains one carbon–carbon double bond. The selectivity can be expressed as the ratio [stearic acid (C18:0)/total TFA] and calculated using Eq. (2) (Fogler, 2006):

$$\text{Trans selectivity} = \frac{\text{No. of moles of desired product}(C18:0)\text{ formed}}{\text{No. of moles of undesired product}(\text{TFA})\text{ formed}} \tag{2}$$

Eq. (3) (Fernandez et al., 2007; Ortiz et al., 1994) was used for estimating of the effect of reaction temperature on the mass transfer coefficient (k_s):

$$(k_s \cdot d_p/D_i)^2 = 16 + 4.84\left(g \cdot d_p^3 \cdot ((\rho_{cat} - \rho_l)/18\mu D_i)\right)^{2/3} \tag{3}$$

3. Results and discussion

3.1. Reaction conversion

Effects of hydrogenation temperature on reaction conversion as indicated by changes in the PMF's di and mono unsaturated fatty acids are illustrated in the results.

Set of hydrogenation experiments were conducted in the temperatures range 99–149 °C in order to study the effect of temperature on the extent of hydrogenation and the corresponding fatty acid profiles. Contents of C18:2, C18:1, C18:0 and trans-fatty acids (TFA) in the PMF samples at the beginning and the end of hydrogenation processes under temperatures ranged from 99 to 149 °C are shown in Table 3. Arithmetic calculations of reaction conversion (X) expressed as percentiles and selectivity as the increase of C18:0 to the increase of TFA concentrations ratio were also presented.

It can clearly be noticed from Table 3, stearic acid (C18:0) content increased in inverse proportion to linoleic acid (C18:2) content whenever hydrogenation temperature increased. Oleic acid (C18:1) content was also altered when hydrogenation temperature increased, but not in inverse relation to the same extent that linoleic acid (C18:2) had with stearic acid (C18:0). Looking over the reaction conversion values (X), would also allow drawing similar inference however percentile values of reaction conversion at temperatures 99 and 130 °C were found to be considerably lower, when compared to those at temperatures 137 and 149 °C.

It is apparent that reaction conversion (X) is directly proportional to hydrogenation temperature. It might be reasonable to infer that the increase in reaction conversion indicated by the percentile values for each PMF sample is attributable to the increase in hydrogen solubility influenced by hydrogenation temperature. Such inference is supported by results of similar research works published previously (Fernandez et al., 2007; Savchenko and Makaryan, 1999;

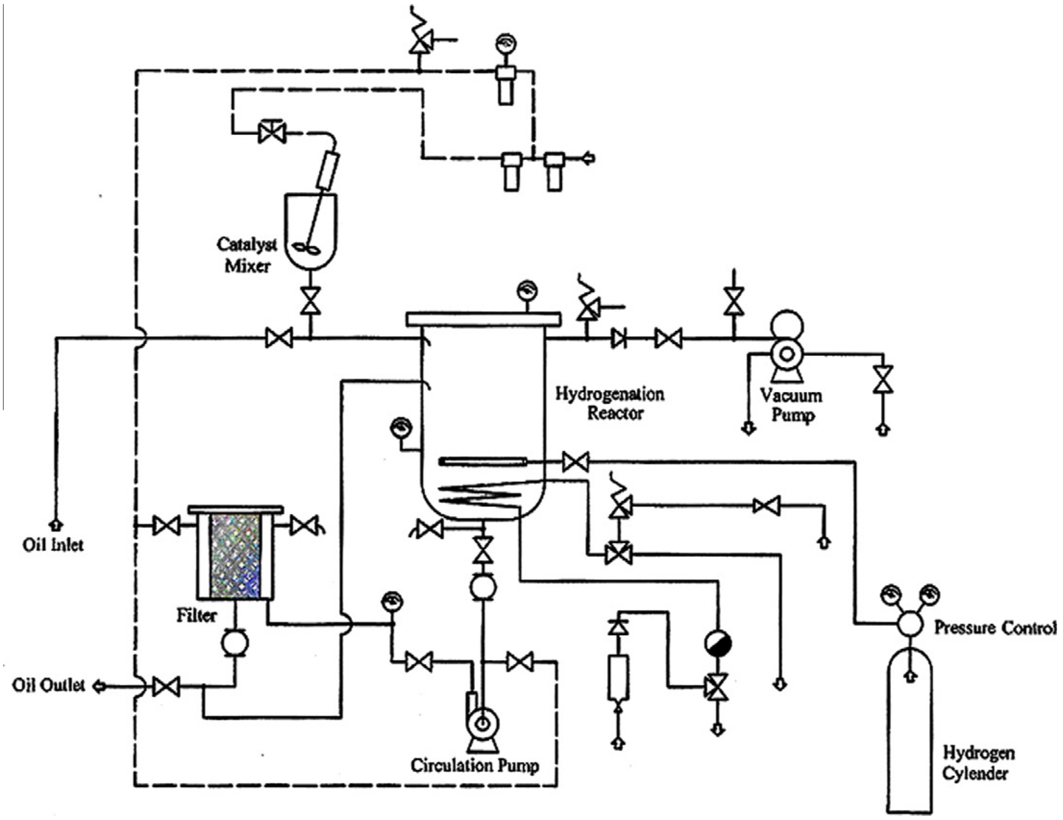

Figure 1 Hydrogenation pilot plant (Armfield).

Table 3 Effects of varying hydrogenation temperature on contents of C18:2, C18:1, C18:0 and total TFA in PMF.

Sample code	Temperature (°C)	Content (mole/15 kg PMF)					Conversion (%)	Selectivity C18:0/TFA
			C18:0	C18:1	C18:2	TFA		
32	99	F_i	2.43	20.92	5.13	0.21	5.16	1.48
		F_o	3.45	20.7	4.44	0.9		
		D	1.02	0.22	0.69	0.69		
22-A	130	F_i	2.43	20.92	5.13	0.21	17.70	0.84
		F_o	5.54	21.2	2.24	3.93		
		i	3.11	0.28	2.89	3.72		
27-A	137	F_i	2.43	20.92	5.13	0.21	23.37	0.77
		F_o	6.38	20.8	1.55	5.31		
		D	3.95	0.12	3.5	5.1		
29-A	149	F_i	2.43	20.92	5.13	0.21	32.03	0.78
		F_o	8.12	20.34	0.43	7.54		
		D	5.69	0.58	4.7	7.33		

F_i is the initial content, F_o is the out content and D is the difference.

Fillion et al., 2002), where they all agreed on that: "hydrogenation reaction in oils subjected to either conventional or industrial hydrogenation is strongly limited by the low hydrogen solubility and its mass transfer from the bulk to the catalyst surface".

It is also clearly noticeable that TFA content of each PMF sample was remarkably increased whenever hydrogenation temperature increased. It can also be observed that the rates of TFA content in the PMF samples hydrogenated at temperature ranges 130–137 °C, and 137–149 °C, when calculated as mole/15 kg PMF/°C, were found to be 0.197 and 0.186, respectively. Such rates were considerably higher than the rate 0.098 mole/15 kg PMF/°C, calculated for the PMF sample hydrogenated at temperature range 99–130 °C. Hence, increasing hydrogenation temperature promotes trans-isomerization and consequently leads to increase in TFA content in the hydrogenated PMF samples. These results were in a good agreement with what (Santana et al., 2007; Singh et al., 2009), found regarding the relation between hydrogenation temperature and TFA formation in hydrogenated vegetable oils, where they indicated that TFA formation is an essential function of temperature.

The increases in the amount of TFA formation have also been associated with changes in the rate of reaction conversion. Fig. 2 represents the relation of total TFA content as a function of reaction conversion in the hydrogenated PMF samples. Along with the increases in rates of reaction conversion, an increase for the demand of hydrogen at the catalyst surface is required. However in case of temperature is increased while other variables are kept constant, hydrogen supply onto the catalyst surface might not be sufficient to complete the saturation. Hence, an increase in TFA rate formation would occur (Fernandez et al., 2007). The values calculated from the increase of C18:0 to the increase of TFA concentrations ratio (C18:0/TFA) shall reflect the extent of C18:0 formation as a favored product over TFA in the PMF samples. Though a high selectivity value was observed (Table 3) for the PMF sample hydrogenated at 99 °C, a sudden decrease in the selectivity value was noticeably found for the PMF sample hydrogenated at 130 °C. A gradual decrease in selectivity value for each of the other PMF samples hydrogenated at either 137 or 149 °C was noticed, however both values were found to be almost the same.

A high selectivity value in any of the PMF sample subjected to hydrogenation at specified temperature means that the rate of formation of the desired product, which is C18:0 is higher than the rate of TFA formation. Therefore, under hydrogenation condition set for hydrogenating the PMF samples in this study, the selectivity value of the PMF sample hydrogenated at 90 °C was found high. The decreases in selectivity values for the PMF samples hydrogenated at temperatures 130, 137 and 149 °C are attributed to the increases in TFA formed at such high temperatures (Singh et al., 2009), hence denominator value of the C18:0/TFA ratio was increasing, and lower selectivity values were observed.

3.2. *Effect of temperature on mass transfer resistance*

The hydrogenation of PMF over a nickel catalyst is a three-phase system. Two reactants diffusion processes are occurring in a heterogeneous reaction: mass transfer from the bulk to the external surface of the catalyst, and the reactants diffusion from the external surface into the pores of the catalyst (Fernandez et al., 2007).

To understand and interpret the effect of reaction temperature on the reaction conversion, it was needed to evaluate the effect of temperature on the mass transfer resistance from the hydrogen bubble to the nickel catalyst surface.

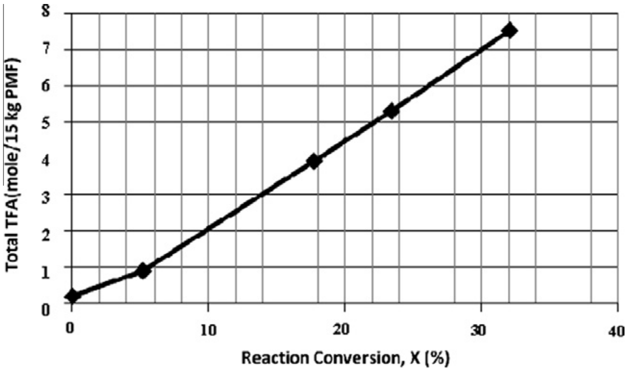

Figure 2 Total TFA content as a function of reaction conversion.

3.2.1. *Diffusion in the gas-phase*

The diffusion of hydrogen in the gas bubble has not been considered since the diffusivity in gases is much greater than in liquids (Fernandez et al., 2007). All experiments were made with pure hydrogen constantly bubbling in the oil, and the vapor pressure of the PMF is negligible at the reaction temperatures of 99–149 °C (Yuan et al., 2005).

3.2.2. *Mass transfer in the gas–liquid Interphase*

Some researchers (Fernandez et al., 2007; Fogler, 2006; Veldsink et al., 1997) proposed to estimate the magnitude of the different mechanisms for mass transport by plotting the reciprocal of the hydrogenation rate ($C_{H2}/r_{H2)}$ versus the inverse of the catalyst mass ($1/m$) according to the Eq. (4):

$$C_{H2}/r_{H2} = [1/k_b \cdot a_b] + 1/m(1/k_c \cdot a_c) + (1/k \cdot \eta) \qquad (4)$$

In agreement with this equation, the intercept at the y-axis is associated with the magnitude of the mass transfer resistance between the gas and the liquid, whereas the slope depends on combined resistance to the internal diffusion, reaction, and external diffusion (Fernandez et al., 2007; Fogler, 2006).

Two sets of experiments were done using different nickel catalyst concentrations at 99 and 130 °C in order to study the effect of temperature on mass transfer resistance of hydrogen molecule from the hydrogen bubble to the catalyst surface. The results of the two sets of experiments have been represented in Table 4. The graph C_{H2}/r_{H2} versus $1/m$ for experiments at 99 and 130 °C is shown in Fig. 3. In order to make possible comparison of hydrogenation at different catalyst load, two similar catalyst loads were used at each reaction temperature. The value of C_{H2}/r_{H2} for $1/m = 1.75\,\text{m}^3$ (PMF)/kg Ni at 99 °C was estimated by extrapolation. As shown in Fig. 3 a similar tendency is seen between 99 and 130 °C.

The volumetric coefficient ($k_b a_b$) was measured using a relatively high dose of nickel catalyst because the data at high catalyst loading are more accurate due to that the impurities present in natural oils have more pronounced effect on the observed reaction rate at low catalysts loading (Jonker, 1999). The $k_b a_b$ was $0.0095\ \text{s}^{-1}$ for the reaction at 99 °C, whereas at 130 °C it was $0.024\ \text{s}^{-1}$. This shows that the higher the temperature, the higher the volumetric hydrogen–PMF mass transfer coefficient reached. This result clarify that the volumetric mass transfer coefficient was increased around 140% by increasing the reaction temperature from 99 to 130 °C.

The estimated values of combined resistance to the internal diffusion, reaction, and external diffusion at 99 and 130 °C were 1.1 ($\text{m}^3\,\text{kg}^{-1}\,\text{Ni}\,\text{min}^{-1}$) and 0.32 ($\text{m}^3\,\text{kg}^{-1}\,\text{Ni}\,\text{min}^{-1}$), respectively. It can be concluded that the combined resistance has been reduced 3.44 times by increasing the reaction temperature. The decreasing in the rate of resistances might be attributed to: increase in the reaction rate because the temperature has a major influence on rate of reaction and increase in mass transfer coefficient (k_s) for the hydrogen molecules from the bulk PMF to the catalyst surface, because the temperature has a minor effect in the PMF–nickel catalyst mass transport (Fogler, 2006).

3.2.3. *Transport in the liquid–solid film*

The coefficient of hydrogen mass transfer in the PMF–Ni film was estimated using the Eq. (3).

The effect of reaction temperature on the coefficient of hydrogen mass transfer (k_s) in the PMF–Ni film as indicator

Table 4 Results to study the effect of temperature on mass transfer resistance.

Sample code		38	39	26	21	32
Temperature (°C)		130	130	130	99	99
Ni concentration, m (kg Ni/m^3 (PMF)		0.168	0.304	0.563	0.173	0.346
Composition (k mole/ batch PMF) $\times 10^{-3}$						
C18:0	F_i	2.43	2.43	2.43	2.43	2.43
	F_o	4.06	5.19	7.17	3.04	3.45
	D	1.63	2.76	4.74	0.61	1.02
C18:1	F_i	20.92	20.92	20.92	20.92	20.92
	F_o	21.07	21.4	20.71	20.69	20.40
	D	0.15	0.48	0.21	0.23	0.52
C18:2	F_i	5.13	5.13	5.13	5.13	5.13
	F_o	3.58	2.33	0.96	4.78	4.44
	D	1.55	2.8	4.17	0.35	0.69
Utilized hydrogen (k mol/batch) $\times 10^3$		2.95	5.12	8.55	0.93	1.9
Conversion, X (%)		9.94	16.44	27.35		5.16
Rate of reaction, $r \times 10^3$ (k mol H_2/min m^3 PMF) $\times 10^3$		2.33	4.03	6.74	0.75	1.54
H_2 Concentration, C_{H2} (k mol (H_2)/m^3 PMF) $\times 10^3$		4.72	4.72	4.72	4.62	4.62
$1/m$ (m^3(PMF)/kg Ni)		5.95	3.29	1.78	5.87	2.90
C_{H2}/r_{H2} (min)		2.03	1.17	0.70	6.14	3.00

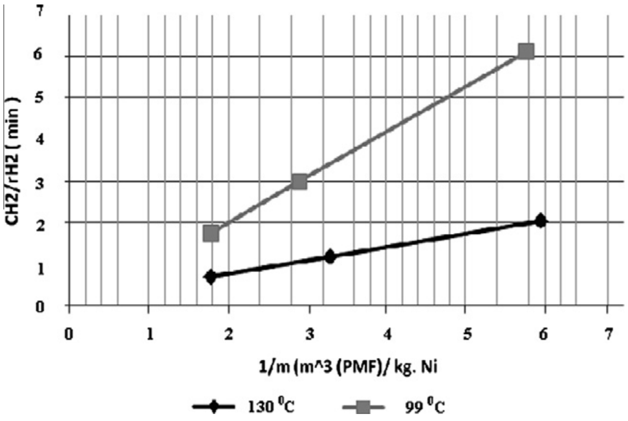

Figure 3 Effect of temperature on mass transfer resistances.

for its effect on the mass transfer rate was estimated. This effect on k_s was might correlated with the effect of temperature on hydrogen diffusivity in the PMF, bulk density of the PMF and viscosity of PMF.

Mass transfer coefficient (k_s) from the bulk PMF to the catalyst surface in each sample hydrogenated at specified hydrogenation temperature was estimated and the results are shown in Table 5. It can be clearly seen that there is a proportional increase in mass transfer coefficient whenever hydrogenation temperature is increased. Such increase in mass transfer coefficient in the PMF samples might be attributable to decrease in oil viscosity resulted from hydrogenation temperature increase.

Table 5 Effect of temperature on mass transfer coefficient (K_s).

Sample code	Temperature (°C)	Mass transfer coefficient (k_s) (m/s) $\times 10^3$
32	99	3.216
22-A	130	3.73
27-A	137	4.04
29-A	149	4.36

It is apparent from Table 5 that the increases in hydrogen mass transfer coefficient are proportional to the increases in hydrogenation temperatures applied for hydrogenating the PMF samples. Such result confirms what previously has been found in this study regarding effects of hydrogenation temperature on reaction conversion.

It can be concluded that in parallel with reaction temperature increase, hydrogen mass transfer was increased, catalyst activity was enhanced and then reaction conversion was increased (Piqueras et al., 2006; Fernandez et al., 2007).

4. Conclusions

Hydrogenation of palm mid fraction was studied using nickel-based catalyst at different reaction temperatures. The hydrogenation temperature is affecting the fatty acid composition and TFA formation in the PMF. The reaction conversion and selectivity has increased with increase in temperature. The limits of TFA which were formed at different levels of the hydrogenation parameters were low content (lower than 15%). This property makes hydrogenated PMF healthier than the hydrogenated soft oils for the application of biscuit, confectionary and baking industry. Increase in hydrogenation temperature is reducing both the resistance to hydrogen diffusion from hydrogen bubble to the PMF and the combined resistance to the internal diffusion, reaction, and external diffusion. The mass transfer coefficient for the hydrogen in the PMF–Ni film was estimated. It was increasing with increase in hydrogenation temperature.

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