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Optimization of the Ring Opening of Epoxidized Palm Oil using D-Optimal Design

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In the presence of a catalyst, *p*-toluenesulfonic acid (PTSA), the ring of epoxidized palm oil (EPO) was opened using oleic acid (OA). The optimization effects of different process variables including the mol ratio of EPO/OA, reaction temperature, PTSA percentage and reaction time was performed by response surface methodology (RSM). To assess the effects of process variables and interactions among them, a D-optimal design was used as an RSM tool to acquire the maximum response value. The following are the optimum conditions achieved at the reaction time of 4.73 h in the RSM study: 1.02% PTSA, 3 EPO/OA mol ratio and 119.14 ºC reaction temperature. These conditions resulted in 84% yield, 0.041% oxirane oxygen content (OOC), 59.4 mg/g iodine value (IV), and118.7 mg/g hydroxyl value (HV). The results are in a excellent agreement with the values predicted using a regression model.

Keywords: Crude palm oil, D-optimal design, Optimization, Ring opening reaction, Biolubricant.

INTRODUCTION

Nowadays, plant oils are used as green raw materials in many area of industries, due to increasing concerns over the use of petroleum-based products which cause progressive reduction of fossil fuels and concerns over the impact on the environment [1,2]. Plant oil-derived renewable products have inferior oxidation stability and low-temperature operability compared to petroleum based products. Chemical modification of the unsaturation in fatty acids such as acylation, metathesis, hydroxylation, oxidative cleavage, carboxylation and epoxidation can provide products with improved oxidation stability and low-temperature properties [3,4]. Epoxide can be reactive intermediates that can be converted to other functional groups through ring-opening reactions due to high reactivity of the oxirane ring [5].

The oxirane ring opening reaction can be carried out using an epoxy moiety; and the opening reaction takes place through the cleavage of the carbon-oxygen bonds, which can be initiated achieved by either using nucleophiles or electrophiles or nucleophiles, or is catalyzed by using either bases or acids or bases. The epoxides can react with different various nucleophiles to produce compounds such as alcohols, diols, alkoxyalcohols, diols, hydroxy esters, *N*-hydroxyalkylamides, hydroxy esters, mercaptoalcohols, amino-alcohols, mercaptoalcohols, *etc*. Amongst these, hydroxy esters are generaly used as biolubricants, polyurethane foams, bio-lubricants and or casting resins. Various carboxylic acids can be used to modify the physico-chemical properties of the hydroxyl ester derived biolubricants [6].

Protonation with solid acid catalysts facilitates the nucleophilic addition of carboxyl groups to epoxide centres [7]. The acid catalysis of epoxides is beneficial for hydroxy ester preparation. *p*-Toluenesulfonic acid (PTSA) as catalyst is used preferably in reactions because it leads to the maximum opening of epoxy rings and causes no side reaction [8]. Ring opening can produce branching groups, interfering with macrocrystalline structure generation in low-temperature applications and providing improved fluidity to plant oil. Plant oils modified with chain branching exhibit superior physico-chemical properties and are the potential candidates for biolubricant use [9].

In this study, the epoxidized palm oil (EPO) oxirane ring was opened by conducting the nucleophilic addition of oleic acid (OA) in the presence of *p*-toluenesulfonic acid (PTSA), which is a homogenous acid catalyst. This study investigated the influence of different reaction variables including the mol ratio of EPO/OA, reaction temperature, PTSA percentage and reaction time and interactions among these variables on EPO

ring opening. D-optimal was employed to realize the effect of these variables by performing the analysis of variance (ANOVA) to obtain the optimal reaction conditions. The oxirane oxygen content (OOC), iodine value (IV) and hydroxyl value can be employed to determine the oxirane ring opening results.

EXPERIMENTAL

The crude palm oil was procured from Sime Darby Sdn. Bhd., Selangor, Malaysia. Epoxidized palm oil (EPO) was obtained *via* epoxidation of palm oil with performic acid. Oleic acid (90%) were purchased from Sigma-Aldrich Chemical Co. Inc. (USA). Ethyl acetate, *p*-toluenesulfonic acid (PTSA), sodium hydrogen carbonate, sodium chloride and sodium sulphate were purchased from Systems & Industrial Chemicals Sdn Bhd.

Epoxidized ring opening reaction: For ring opening, epoxidized palm oil (EPO) and oleic acid (OA) were placed in a three-neck round 250 mL bottom flask equipped with a thermometer, mechanical stirrer and reflux condenser. Subsequently, *p*-toluenesulfonic acid (PTSA) was added into this mixture. The resulting mixture was heated with continuous stirring (900 rpm) by using a magnetic stirrer. Heating was terminated after the completion of the reaction. The product was neutralized using ethyl acetate, sodium hydrogen carbonate and sodium chloride solutions. Anhydrous sodium sulphate was added to the product and then the product was stored overnight. The product was filtered using Whatmann No. 1 filter paper and the solvent was removed by employing a rotary evaporator at 70 ºC.

Experimental design and statistical analysis: The reaction of ring opening comprised 25 experiments and was designed according to a D-optimal design. Table-1 presents different EPO/OA mol ratios, reaction temperature, PTSA percentage, and reaction time obtained using the D-optimal design. Variables including EPO/OA mol ratios (w/w, X_1), reaction temperatures (ºC, X_3), PTSA percentage (%, X_2) and reaction time (h, X_4) were used to investigate the effects of these variables on the opening of the ring. Table-1 presents the extent of the variables of ring opening for low (-1), medium (0) and high (+1) levels of these variables. To predict process response as the function of independent variables, regression coefficients (R^2) were determined. These variable interactions were employed to comprehend system behaviours [10]. A mathematical relationship between process variables and responses was calculated using a quadratic polynomial (eqn. 1):

$$Y = \beta_0 + \Sigma \beta_i x_i + \Sigma \beta_{ii} x_i^2 + \Sigma\Sigma \beta_{ij} x_{ixj} \quad (1)$$

where β_0; β_i; β_{ii} and β_{ij} are constant, linear, square and interaction regression coefficient terms, respectively and x_i and x_j are independent variables. Analysis of variance (ANOVA) was carried out to estimate the effects of process variables and their possible interaction effects on the maximum oxirane oxygen content (OOC) in the response surface regression procedure [7,10]. The goodness and best fit of the model was evaluated by a regression coefficient R^2 [10].

The unsaturation of oil was determined by iodine value test using Wijs method (PORIM method), while the oxirane oxygen content (OOC) was analyzed by direct method using hydrobomic acid solution in acetic acid as prescribed in official method AOCS Cd 9-57 [11].

TABLE-1
PARAMETERS AND LEVELS FOR d-OPTIMAL DESIGN FOR RING OPENING REACTION

Independent variables	Factor	Variable levels		
	X_i	-1	0	+1
EPO/OA (mol)	X_1	1	2	3
PTSA (%)	X_2	1	2	3
Temperature (°C)	X_3	110	120	130
Time (h)	X_4	3	4	5

RESULTS AND DISCUSSION

Addition of several nucleophilic reagents to oxirane rings can lead to ring opening. The ester branching groups formed through esterification based on oxirane ring opening can effectively achieve any desired molecular spacing [12]. In this work, the oxirane ring opening of EPO in the presence of PTSA, a homogenous acid catalyst, through the nucleophilic addition of oleic acid. Most groups of the oxirane ring were opened and converted into ester bonds in molecules having a hydroxyl group [13].

Optimization study of ring opening reaction using D-optimal design was carried out. The design is used to obtain 25 design points with four factors. All the 25 designed experiments have been tabulated in Table-2. Different EPO/OA mol ratio, different percentage of PTSA, different reaction temperature and different reaction time were evaluated in order to evaluate the impact on the responses such as yield %, iodine value (IV) (mg/g), oxirane oxygen content (OOC) % and hydroxyl value (HV) (mg/g) after ring opening reaction.

Based on Table-2, the lowest OOC% obtained was 0.001% at 3.00 EPO/OA mol ratio, 2% PTSA, 130 ºC reaction temperature and 5 h reaction time. This conditions shows a high increment of yield (81.3%), highest IV (60.7 mg/g) and HV of 122 mg/g. The quadratic polynomial equations was obtained from the experimental data to predict the optimal results as shown below in terms of coded variables:

Yield = $77.50 + 8.92A - 0.96B + 0.24C + 0.38D - 3.34A^2 - 0.15B^2 - 1.55C^2 + 0.034D^2 + 0.64AB + 0.32AC - 0.30AD + 0.72BC - 0.23BD - 0.85CD$

Iodine value (IV) = $58.48 + 7.07A + 1.87B - 1.96C + 2.48D - 10.51A^2 - 1.49B^2 + 0.82C^2 + 4.36D^2 - 2.81AB + 2.10AC - 2.00AD - 0.89BC - 0.94BD - 0.46CD$

Oxirane oxygen content (OOC) = $0.042 - 0.49A - 0.19B + 0.13C - 0.19D + 0.68A^2 + 0.090B^2 + 0.046C^2 - 0.33D^2 + 0.27AB - 0.18AC + 0.22AD + 0.22BC + 0.14BD + 0.055CD$

Hydroxyl value (HV) = $117.15 + 12.33A + 2.21B - 3.71C + 4.52D - 15.85A^2 - 1.42B^2 + 1.38C^2 + 5.08D^2 - 4.85AB + 2.46AC - 2.94AD - 1.88BC - 1.82BD + 1.41CD$

Tables 3-6 present the quadratic regression coefficients obtained using the least squares method to predict quadratic polynomial models for determining yield % (Y_1), IV mg/g (Y_2), OOC % (Y_3), and HV % (Y_4). Generally, important process variables are determined on the basis of the F- or P-value (error probability value or 'Prob > F' value). The 'Prob > F' values of < 0.05 indicate significant model terms. The lower was the

TABLE-2
EXPERIMENTAL RUNS FROM d-OPTIMAL DESIGN ARRANGEMENT AND RESPONSES FOR RING OPENING REACTION

Run No.	Variables levels (X)				Responses (Y)			
	EPO/OA[a] mol ratio (mol, X_1)	% PTSA[b] (%, X_2)	Temperature (°C, X_3)	Time (h, X_4)	Yield (%, Y_1)	Iodin value (mg/g, Y_2)	OOC[c] (%, Y_3)	Hydroxyl value (mg/g, Y_4)
1	3.00	1.00	110.00	5.00	82.53	59.9	0.037	124.80
2	2.00	1.50	120.00	4.00	77.90	58.0	0.130	116.70
3	3.00	3.00	130.00	3.00	83.10	57.9	0.067	110.50
4	2.00	1.00	130.00	3.00	76.50	55.5	0.220	106.30
5	1.00	1.00	110.00	3.00	64.00	37.2	2.000	83.73
6	3.00	1.00	120.00	3.00	82.90	57.1	0.021	119.78
7	3.00	2.00	120.00	4.00	83.40	53.5	0.250	110.45
8	2.00	2.00	110.00	4.00	75.67	60.7	0.010	123.38
9	3.00	1.00	130.00	4.00	81.60	57.5	0.056	117.69
10	3.00	3.00	110.00	5.00	80.38	57.0	0.030	119.40
11	3.00	1.00	110.00	5.00	83.51	60.9	0.033	119.50
12	3.00	3.00	110.00	5.00	80.55	60.0	0.029	116.60
13	1.00	3.00	130.00	5.00	62.20	47.1	0.860	99.00
14	3.00	2.00	110.00	3.00	80.05	60.1	0.023	120.15
15	2.00	3.00	120.00	4.00	76.60	60.3	0.017	118.60
16	1.00	2.00	130.00	3.00	64.00	39.0	1.650	82.68
17	1.00	3.00	110.00	3.00	60.00	49.5	0.330	104.45
18	1.00	2.00	110.00	5.00	65.48	55.6	0.170	107.90
19	1.00	1.00	130.00	5.00	64.40	42.8	0.870	93.40
20	2.00	2.00	130.00	4.00	76.10	56.4	0.130	116.10
21	3.00	3.00	130.00	3.00	83.55	57.8	0.075	112.90
22	1.00	1.00	130.00	5.00	64.50	40.4	1.300	93.90
23	1.00	2.00	120.00	4.00	64.80	40.5	1.150	87.20
24	3.00	2.00	130.00	5.00	81.30	60.7	0.001	122.00
25	1.00	1.00	110.00	3.00	64.50	37.3	1.980	83.00

Notes: [a]Epoxidized palm oil/oleic acid; [b]*p*-toluenesulfonic acid, [c]Oxirane oxygen content (OOC)

TABLE-3
REGRESSION COEFFICIENTS OF THE PREDICTED QUADRATIC POLYNOMIAL MODEL FOR THE RESPONSE VARIABLES OF THE YIELD (%)

Variables	Coefficients (β), Yield % (Y_1)	F value	P-value (Prob > F)	Notability
Linear				
X_1	8.92	10348.97	< 0.0001	***
X_2	-0.96	116.64	< 0.0001	***
X_3	0.24	7.56	0.0205	**
X_4	0.38	15.53	0.0028	***
Quadratic				
X_{11}	-3.34	213.75	< 0.0001	***
X_{22}	-0.15	0.86	0.3764	
X_{33}	-1.55	45.42	< 0.0001	***
X_{44}	0.034	0.017	0.8996	
Interaction				
X_{12}	0.64	43.34	< 0.0001	***
X_{13}	0.32	10.67	0.0085	***
X_{14}	-0.30	9.31	0.0122	**
X_{23}	0.72	54.11	< 0.0001	***
X_{24}	-0.23	5.37	0.0430	**
X_{34}	-0.85	77.05	< 0.0001	***
R^2	0.9993			
Adj R^2	0.9984			
Pred R^2	0.9949			

Notes: $^{**}p < 0.05$, $^{***}p < 0.01$

TABLE-4
REGRESSION COEFFICIENTS OF THE PREDICTED QUADRATIC POLYNOMIAL MODEL FOR THE RESPONSE VARIABLES OF THE IODINE VALUE (%)

Variables	Coefficients (β), iodine value % (Y_2)	F value	P-value (Prob > F)	Notability
Linear				
X_1	7.07	389.93	< 0.0001	***
X_2	1.87	26.45	0.0004	***
X_3	-1.96	29.79	0.0003	***
X_4	2.48	39.71	< 0.0001	***
Quadratic				
X_{11}	-10.51	126.93	< 0.0001	***
X_{22}	-1.49	4.89	0.0514	
X_{33}	0.82	0.76	0.4038	
X_{44}	4.36	16.28	0.0024	***
Interaction				
X_{12}	-2.81	49.55	< 0.0001	***
X_{13}	2.10	26.85	0.0004	***
X_{14}	-2.00	25.38	0.0005	***
X_{23}	-0.89	5.03	0.0487	**
X_{24}	-0.94	5.34	0.0434	**
X_{34}	-0.46	1.37	0.2691	
R^2	0.9885			
Adj R^2	0.9724			
Pred R^2	0.7751			

Notes: $^{**}p < 0.05$, $^{***}p < 0.01$

P-value, the more significant was the variable; the relative significance raking of all the variance attached to terms was obtained [14].

For yield response (Y_1), X_1, X_2 and X_4 linear terms were substantially significant ($P < 0.01$) and the X_3 linear term was significant ($P < 0.05$). For OOC % (Y_3) and IV mg/g (Y_2)

TABLE-5
REGRESSION COEFFICIENTS OF THE PREDICTED QUADRATIC POLYNOMIAL MODEL FOR THE RESPONSE VARIABLES OF THE OOC %

Variables	Coefficients (β), OOC % (Y_2)	F value	P-value (Prob > F)	Notability
Linear				
X_1	-0.49	298.96	< 0.0001	***
X_2	-0.19	43.78	< 0.0001	***
X_3	0.13	22.08	0.0008	***
X_4	-0.19	39.01	< 0.0001	***
Quadratic				
X_{11}	0.68	84.45	< 0.0001	***
X_{22}	0.090	2.87	0.1211	
X_{33}	0.046	0.38	0.5527	
X_{44}	-0.33	14.75	0.0033	***
Interaction				
X_{12}	0.27	73.24	< 0.0001	***
X_{13}	-0.18	29.67	0.0003	***
X_{14}	0.22	46.91	< 0.0001	***
X_{23}	0.22	46.98	< 0.0001	***
X_{24}	0.14	18.13	0.0017	***
X_{34}	0.055	3.12	0.1080	
R^2	0.9881			
Adj R^2	0.9714			
Pred R^2	0.9120			

Notes: $^{**}p < 0.05$, $^{***}p < 0.01$; OOC = Oxirane oxygen content

TABLE-6
REGRESSION COEFFICIENTS OF THE PREDICTED QUADRATIC POLYNOMIAL MODEL FOR THE RESPONSE VARIABLES OF THE HYDROXYL VALUE (mg/g)

Variables	Coefficients (β), Hydroxyl value % (Y_2)	F value	P-value (Prob > F)	Notability
Linear				
X_1	12.33	303.57	< 0.0001	***
X_2	2.21	9.47	0.0117	**
X_3	-3.71	27.31	0.0004	***
X_4	4.52	33.69	0.0002	***
Quadratic				
X_{11}	-15.85	73.92	< 0.0001	***
X_{22}	-1.42	1.14	0.3110	
X_{33}	1.38	0.55	0.4748	
X_{44}	5.08	5.63	0.0390	**
Interaction				
X_{12}	-4.85	37.78	0.0001	***
X_{13}	2.46	9.39	0.0120	**
X_{14}	-2.94	13.98	0.0039	***
X_{23}	-1.88	5.71	0.0380	**
X_{24}	-1.82	5.14	0.0468	**
X_{34}	1.41	3.22	0.1032	
R^2	0.9832			
Adj R^2	0.9597			
Pred R^2	0.7984			

Notes: $^{**}p < 0.05$, $^{***}p < 0.01$.

responses, all X_1 X_2, X_3 and X_4 linear terms were considerably significant ($P < 0.01$). For HV % response, X_1, X_3, and X_4 linear terms were substantially significant ($P < 0.01$) and the X_2 linear term was significant ($P < 0.05$). The yield % response indicated that X_{11} and X_{33} quadratic terms were extremely significant ($P < 0.01$). For both OOC % and IV mg/g responses, X_{11} and X_{44} quadratic terms were highly significant ($P < 0.01$). For HV mg/g response, X_{11} and X_{44} quadratic terms were substantially significant ($P < 0.01$) and significant ($P < 0.05$), respectively. The yield % response indicated that the interaction terms of all responses were significant, *i.e.* X_{12}, X_{13}, X_{23} and X_{34} interaction terms were substantially significant ($P < 0.01$) and X_{14} and X_{24} interaction terms were significant ($P < 0.05$). For IV mg/g response, X_{12}, X_{13}, and X_{14} interaction terms were considerably significant ($P < 0.01$) and X_{23} and X_{24} interaction term were significant ($P < 0.05$). For OOC % response, X_{12}, X_{13}, X_{14} X_{23}, and X_{24} interaction terms were extremely significant ($P < 0.01$). For HV mg/g response, X_{12} and X_{14} interaction terms were considerably significant ($P < 0.01$) and X_{13} X_{23} and X_{24} interaction term were significant ($P < 0.05$).

The multivariate technique, ANOVA, was used to determine the most favourable reaction conditions. To ensure complete model fit and to perform the analysis of variance on individual model coefficients, lack-of-fit must be estimated [10]. The lack-of-fit is a test for determining model failure to represent the data, which cannot be acquired through random errors [15]. Table-7 presents ANOVA for Y_1, Y_2, Y_3, and Y_4 responses and the F-values for these models are 1062.69, 61.46, 59.30, and 41.79, respectively. The P-values were used to verify the importance of all coefficients [10]. The P-value (Prob > F) corresponding to all responses were considerably small, *i.e.* < 0.0001, which indicated that the regression model for the data on Y_1, Y_2, Y_3 and Y_4 responses was highly significant ($P < 0.01$) with suitable R^2. The F-value of lack-of-fit for all Y_1, Y_2, Y_3, and Y_4 responses indicated that the lack-of-fit was non-significant ($P > 0.05$). The non-significant F-values the of lack-of-fit of 0.6, 1.46, 0.31 and 2.57 for Y_1, Y_2, Y_3 and Y_4, respectively, indicated that relative to a pure error, the lack-of-fit was highly significant. This finding indicated that the response predictions of all the models were sufficient.

TABLE-7
ANALYSIS OF VARIANCE (ANOVA) FOR ALL THE RESPONSES

Variance	Sum of square	Degree of freedom	Mean square	F-values	P-value
Y_1	1731.80	14	123.70	1062.69	< 0.0001
	1.16	10	0.12		
	0.44	5	0.088	0.60	0.7035
	0.73	5	0.15		
Y_2	1669.85	14	119.28	61.46	< 0.0001
	19.41	10	1.94		
	11.52	5	2.30	1.46	0.3441
	7.89	5	1.58		
Y_3	10.10	14	0.72	59.30	< 0.0001
	0.12	10	0.012		
	0.029	5	5.788×10^{-3}	0.31	0.8864
	0.093	5	0.019		
Y_4	437.47	14	316.96	41.79	< 0.0001
	75.85	10	7.59		
	54.61	5	10.92	2.57	0.1616
	21.24	5	4.25		

Model precision is determined using regression coefficients (R^2). The regression coefficient indicates that the accuracy and general capacity of a polynomial model is satisfactory. The value of R^2 is between 0 and 1, and its magnitude order indicates model aptness [16]. For a suitable statistical model, the regression coefficient must be closer to one. Regression coefficients for Y_1, Y_2, Y_3 and Y_4 are 0.9993, 0.9885, 0.9881 and 0.9832, respectively (Tables 3-6), which are close to 1, indicating that 99.93%, 98.85%, 98.81% and 98.32% model behaviour can be interpreted for optimum conditions including high IV, high yield, high HV and low OOC. Furthermore, for each response, only 0.07%, 1.15%, 1.19%, and 1.68% full variance cannot be explained using the model.

The adjusted determination coefcients (R^2) revealed the advantages of the model [13]. For Y_1, Y_2, Y_3 and Y_4, the predicted R^2 values of 0.9949, 0.7751, 0.9120, and 0.7984, respectively, are in reasonable agreement with the adjusted R^2 values of 0.9984, 0.9724, 0.9714 and 0.9597, respectively. It recommends important correlational statistics between the predicted data and remarked values [10]. Thus, this regression model can be satisfactorily used to explain the relationship between response and independent process variables [10,12]. The observed and predicted values are considerably close to each other (Fig. 1).

Influence of various process variables on optimum responses: The effect of interaction between variables on the epoxidation can be illustrated by using the 3-D response surfaces. The significant interaction variables in the fitted models (Tables 3-6) were chosen as the axes (EPO/OA mol ratio X_1, percentage of PTSA X_2, reaction temperature, X_3 and reaction time X_4) for the response surface plots [17].

The influence of EPO/OA mol ratio and percentage of PTSA on yield, IV, OOC and HV was investigated at various mol ratio of EPO/OA and percentage of PTSA (Fig. 2a-d). Increasing of EPO/OA mol ratio and percentage of PTSA have increased the IV and HV. The value of yield increased with increasing EPO/OA mole ratio, but slighly decrease with increase percentage of PTSA. While increasing EPO/OA mol ratio and PTSA% decreased the OOC value. The lowest value of OOC could obtained in high mol ratio of EPO/OA and lower percentage of PTSA [18].

The effect of EPO/OA mol ratio and temperature on yield, IV, OOC and HV was investigated at various mol ratio of EPO/OA and reaction temperature (Fig. 3a-d). Increasing of EPO/OA mol ratio has increased the yield, IV and HV but increasing temperature has decreased IV and HV. The mol ratio of EPO/OA higher than 2.5 showed the decreasing of both IV and HV. While, for OOC value, increasing mol ratio of EPO/oleic acid decrease the OOC until mol ratio of 2, but further increment of mol ratio of EPO/OA would lead to increasing of OOC value. It shows that lowest OOC could be obtained at moderate mol ratio of 2 at lower temperature (110 ºC) [19].

Effect of EPO/OA mol ratio and time on yield IV, OOC and HV can be observed on Fig. 4a-d. The figures showed with the increasing of EPO/OA and time, there were increasing of yield, IV and HV values. Increasing of EPO/OA has decreased the OOC. Lower value of OOC achieved at 2.5 mol ratio of EPO/OA and shorter time (3 h) but further addition of EPO/OA higher than 2.5 showed increasing of OOC [20].

Fig. 5 shows the effect of varying percentage of PTSA and reaction temperature on yield, IV, OOC and HV. Increasing percentage of PTSA has increased the IV and HV and increasing of temperature has decreased these values. It showed different trend with yield response. For OOC value, increasing percentage of PTSA has decreased the OOC to the lowest value [21,22]. Fig. 6 shows the effect of varying percentage of PTSA and reaction time on yield, IV, OOC and HV. Increasing percentage of PTSA and time have increased the IV and HV. Increasing percentage of PTSA has decrease the yield and increasing time has increased yield, while increasing percentage of PTSA has slightly decrease the OOC. The increasing of reaction time could increase the OOC, but further increment of time higher than 4 h would lead to a decline of OOC. The effect of reaction temperature and time only can be seen for yield response (Fig. 7). Fig. 7 showed yield has increased with increasing reaction temperature and time [23].

Conclusion

In this work, the ring opening process of epoxidized palm oil (EPO) was successfully optimized. The interaction between the variables was well explained by quadratic polynomial and ANOVA. The model was also examined for the best fit. The obtained optimum reaction conditions for EPO ring opening were 3.00 EPO/OA mol ratio, 1.02 % PTSA, 119.14 ºC reaction temperature at 4.73 h reaction time. At this optimal condition,

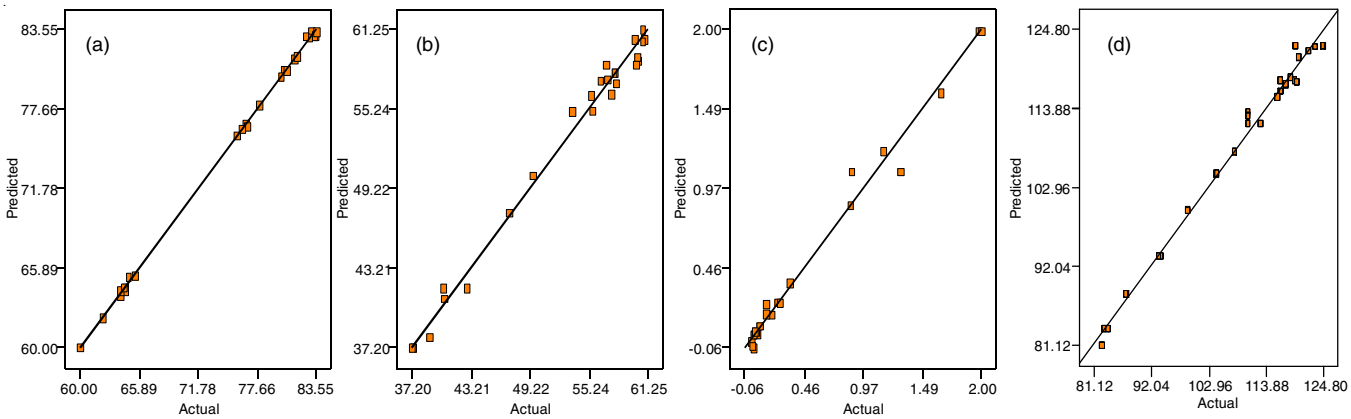

Fig. 1. Actual and predicted plot of (a) yield (b) iodine value (c) oxirane oxygen content (d) hydroxyl value

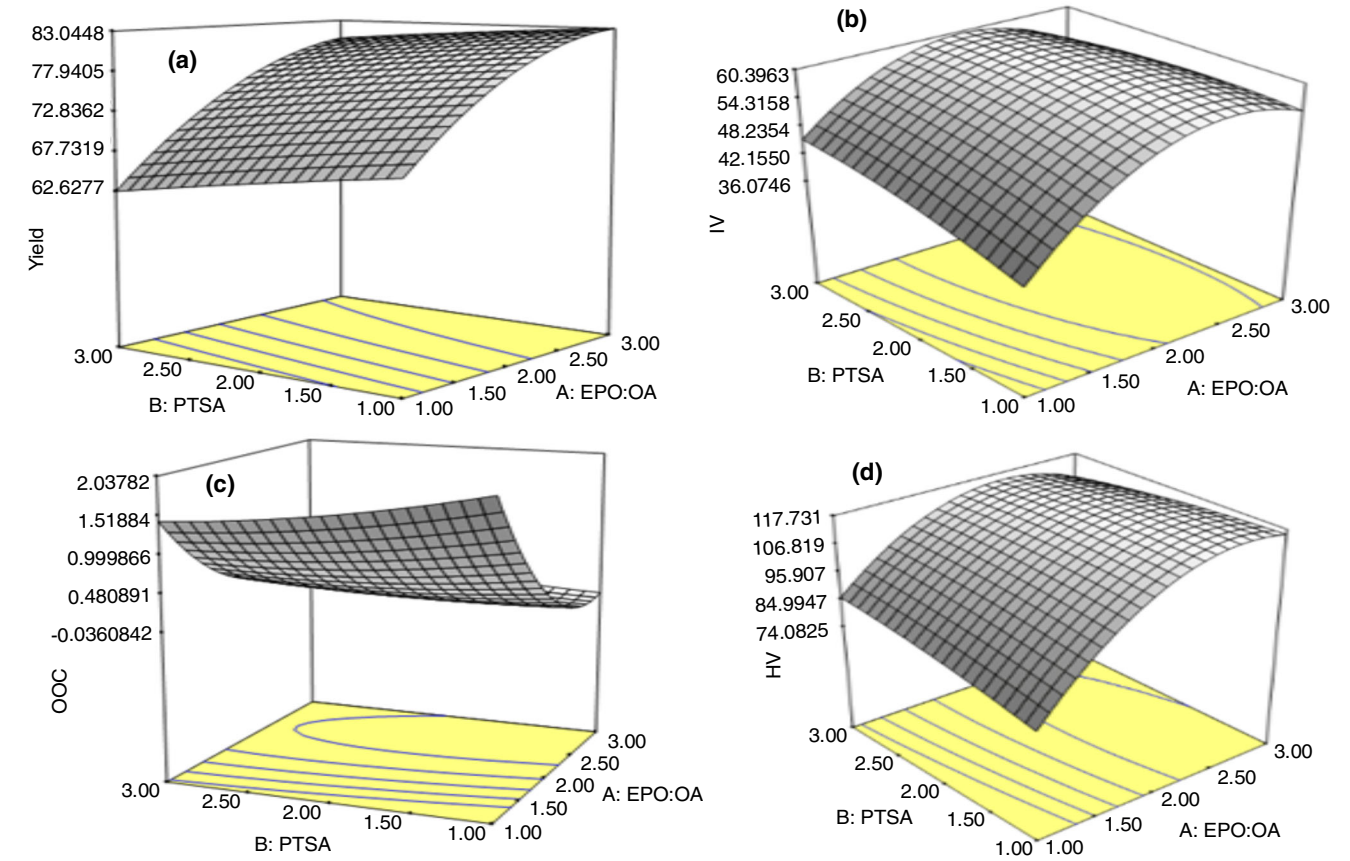

Fig. 2. Effect of the EPO/OA mol ratio and percentage of PTSA on (a) yield, (b) iodine value, (c) oxirane oxygen content (d) hydroxyl value

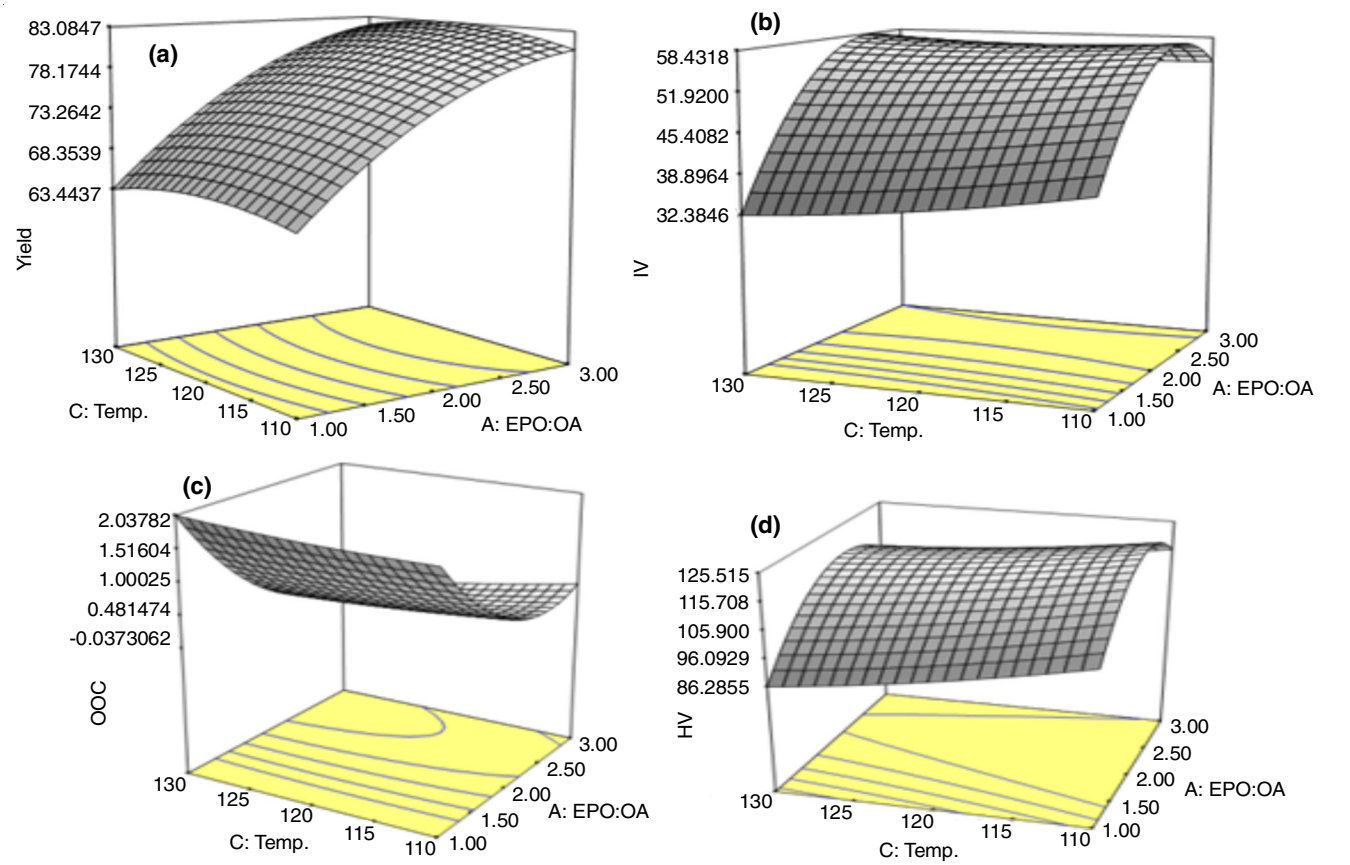

Fig. 3. Effect of the EPO/OA mol ratio and temperature on (a) yield, (b) iodine value, (c) oxirane oxygen content (d) hydroxyl value

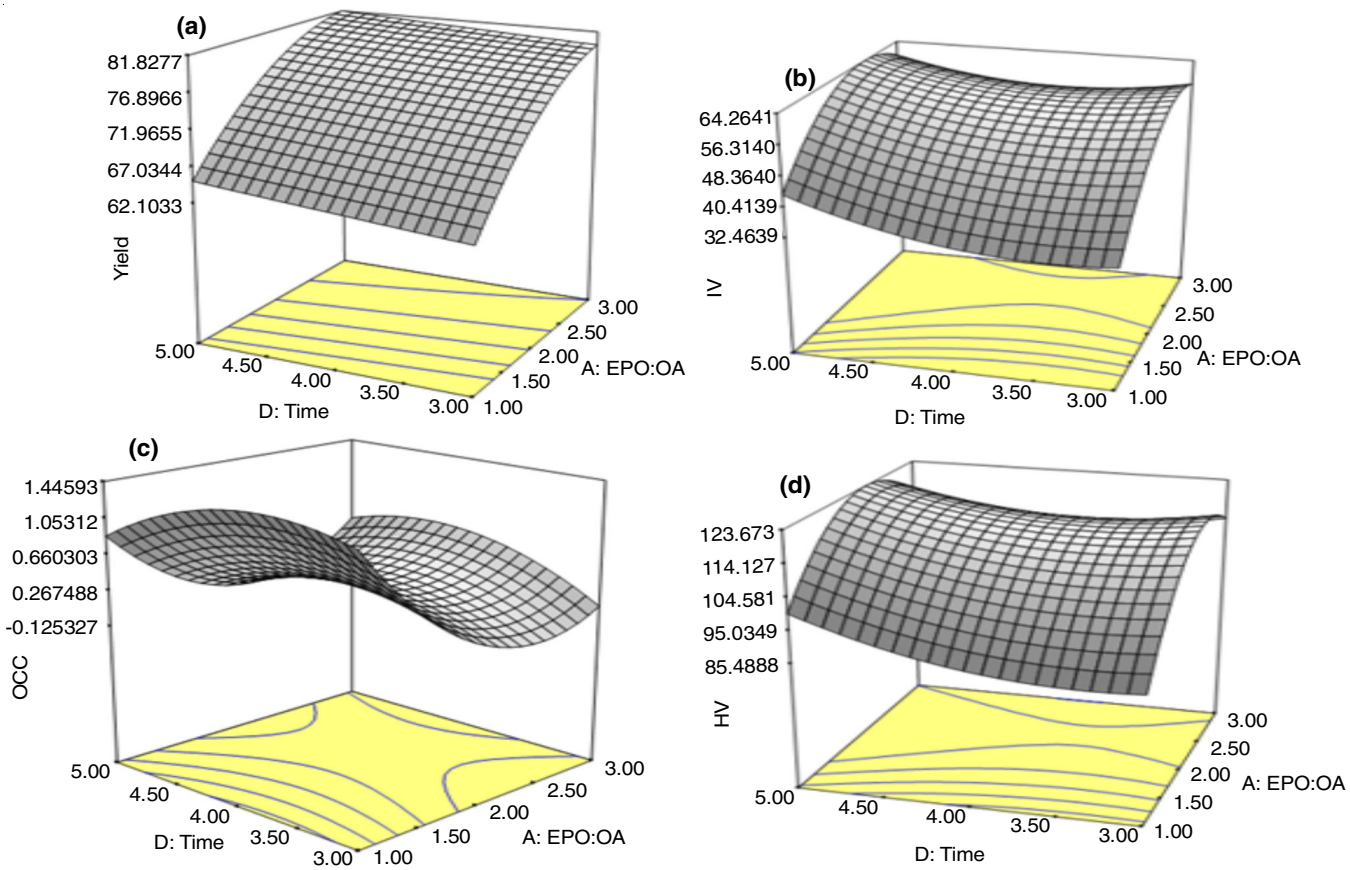

Fig. 4. Effect of the EPO/OA mol ratio and time on (a) yield, (b) iodine value, (c) oxirane oxygen content (d) hydroxyl value

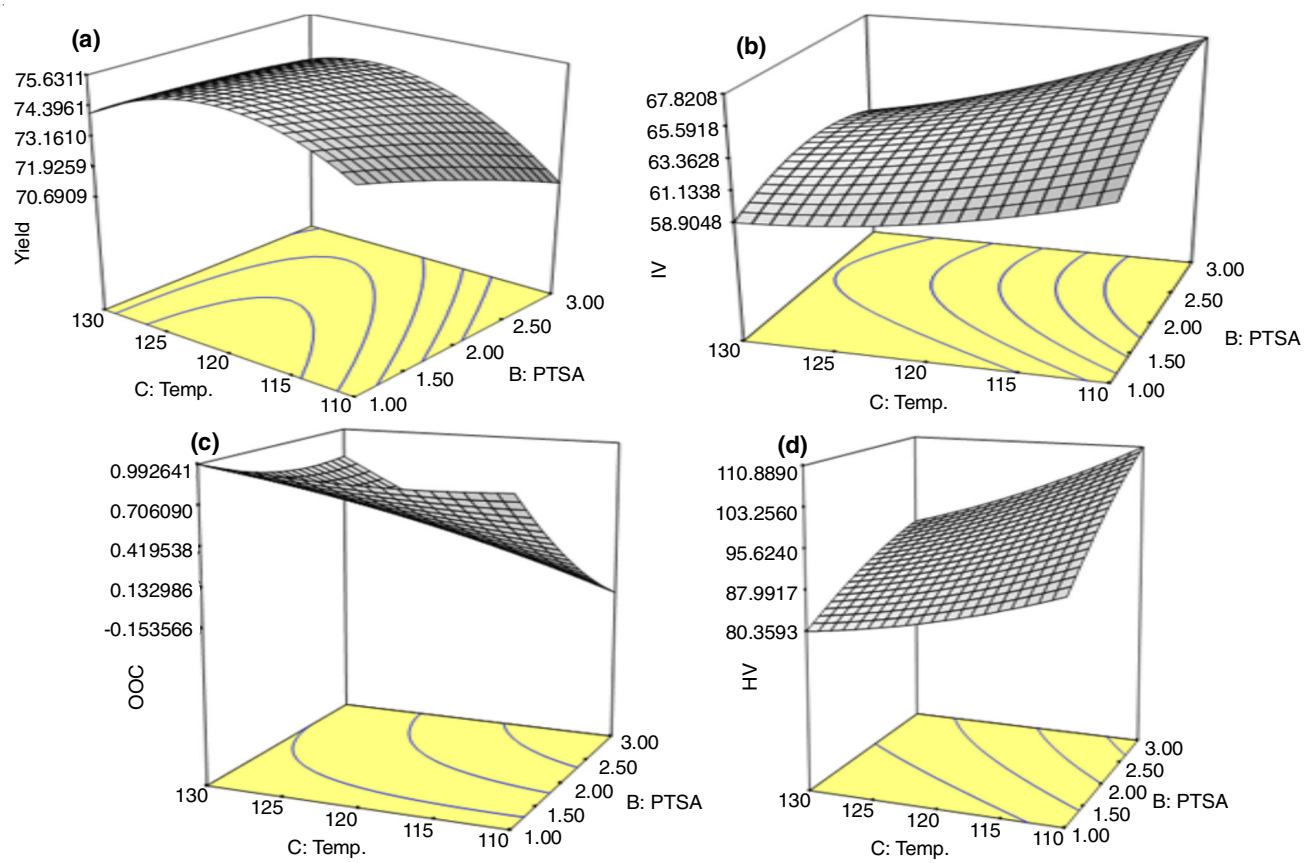

Fig. 5. Effect of percentage of PTSA and temperature on (a) yield, (b) iodine value, (c) oxirane oxygen content (d) hydroxyl value

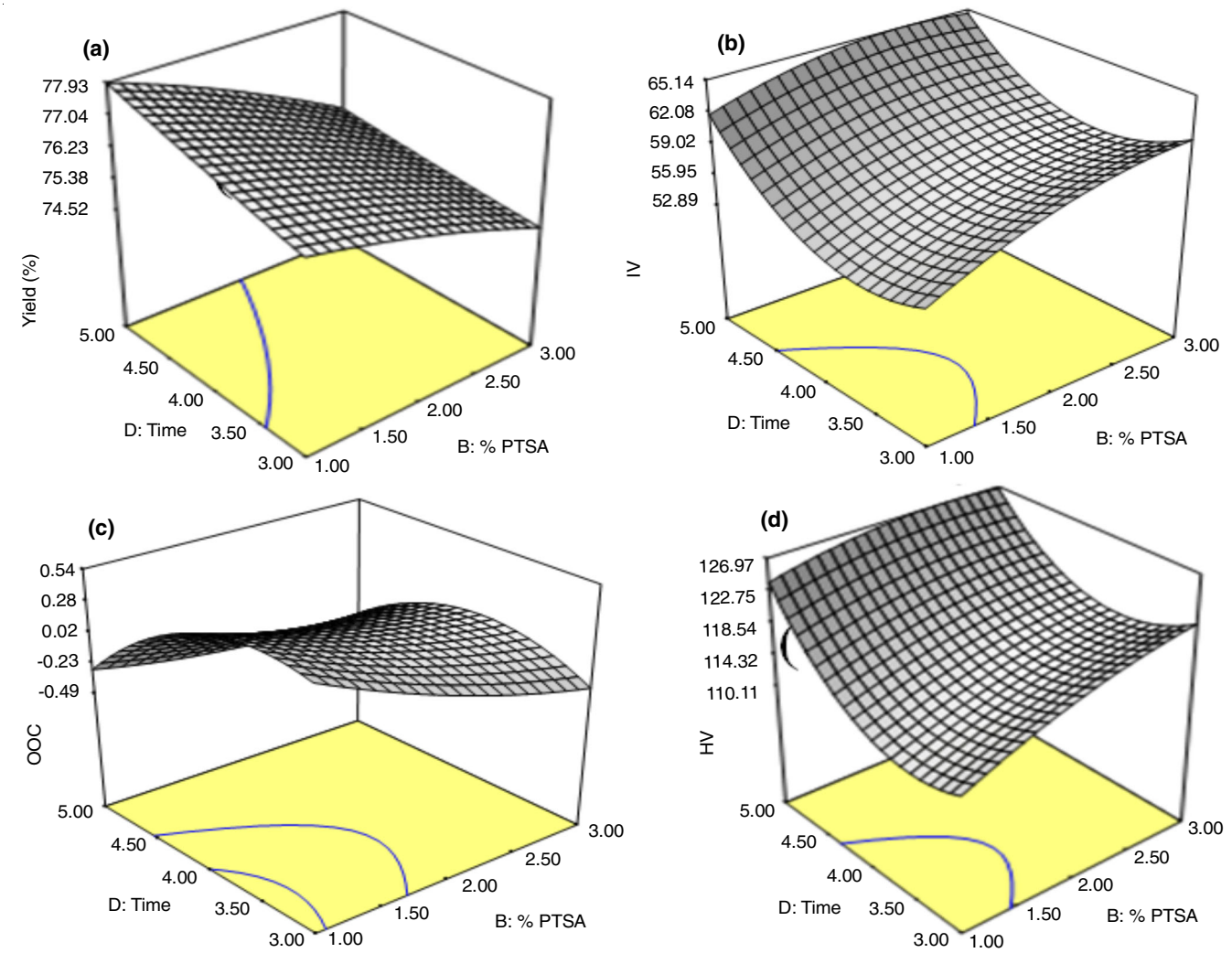

Fig. 6. Effect of percentage of PTSA and time on (a) yield, (b) iodine value, (c) oxirane oxygen content (d) hydroxyl value

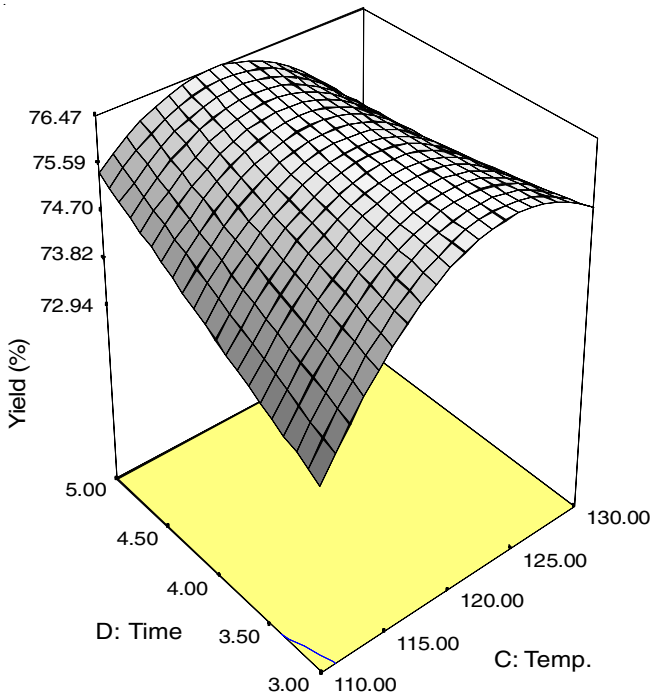

Fig. 7. Effect of temperature and time on the yield

the values of responses were 84% yield, 59.4 mg/g iodine value (IV), 0.041% oxirane oxygen content (OOC) and118.7 mg/g hydroxyl value (HV), which agreed well with the predicted values from the model.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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