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## Preparation of Multi-Walled Carbon Nanotubes Supported Zinc Oxide Catalyst for Transesterification of Kesambi (*Schleichera oleosa*) Oil

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# Preparation of Multi-Walled Carbon Nanotubes Supported Zinc Oxide Catalyst for Transesterification of Kesambi (*Schleichera oleosa*) Oil

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**Abstract.** In this work, multi-walled carbon nanotubes supported zinc oxide (ZnO/MWCNTs) as a heterogeneous catalyst for transesterification had been successfully prepared via the modified Stober-like synthesis method. The effect of calcination temperature during the catalyst preparation towards reaction was investigated. These catalyst was also characterized by X-ray diffraction (XRD) analysis, N<sub>2</sub> adsorption-desorption analysis followed by Brunauer-Emmett-Teller (BET) calculation to determine the total surface area, and scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM-EDX). Transesterification of kesambi (*Schleichera oleosa*) oil also had been done to test the activity of the prepared catalysts. However, the quantity of dispersed zinc oxide (ZnO) on multi-walled carbon nanotubes (MWCNTs) are quite small that is lower than 5 %wt. It produces low conversion of transesterification after 3 hours reaction that is under 15% of conversion.

## 1. Introduction

One of the problems of biodiesel development in Indonesia is the higher production cost than that of petroleum diesel. This problem can be happened due to its production process still utilize homogeneous acid or base catalyst [1]. That conventional process has a lot of problem that is side product and alkaline waste formation in huge amount, and also the complicated separation of catalyst from the product [2]. The other problem on biodiesel production in Indonesia is palm oil utilization as a resource. Palm oil is quite expensive and usually used as food sources called an edible oil. Utilizing palm oil as resources of biodiesel synthesis leads higher production cost and competes with food industry [3].

Non edible oil (NEO) has great potential to be used as a feedstock for biodiesel synthesis because of its low price. However, due to its high free fatty acid (FFA) content, synthesis of biodiesel via conventional process from NEO needs two steps, there is esterification and transesterification [4, 5]. These two steps give an impact on rising energy needs so that increasing its production cost [1]. Moreover, utilizing homogeneous catalyst requires further purification step to separate the catalyst from its product [6]. Addition of unit processes in biodiesel production plant surely increases plant



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investment and production cost. Heterogeneous catalyst development will be the right solution to decrease production cost because it has many advantages, e.g. easy separation from the final product, the catalyst reusability, and environmentally friendly materials [7].

In the previous study, the authors developed several heterogeneous base catalyst, e.g.,  $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$  and double promoted  $\text{CaO}/\text{KI}/\gamma\text{-Al}_2\text{O}_3$  for transesterification of palm oil [3, 7]. The result of this research shows that both of these catalysts have a high activity towards the transesterification reaction especially  $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$  giving 95 – 97 % yield of biodiesel [3]. However, these catalysts are still sensitive to low grade oil (LGO) which has high FFA content like non edible oil. In the other study,  $\text{CaO}/\text{KI}/\gamma\text{-Al}_2\text{O}_3$  was tested to catalyse transesterification of waste cooking oil resulting lower yield of biodiesel at the range of 80 – 83% [5]. The heterogeneous acid catalyst could overcome this problem because it is relatively less sensitive towards FFA than heterogeneous base catalyst [8].

Many researchers had developed solid acid catalysts that can catalyse esterification and transesterification simultaneously for biodiesel synthesis from LGO [4]. This catalyst subsequently named as super acid catalyst because of its strong acid properties [9]. There are many mixed-oxide metal group that had been investigated as super acid catalyst, e.g. titanium oxide ( $\text{TiO}_2$ ) [10], zirconium oxide ( $\text{ZrO}_2$ ) [11], modified zirconium oxide with tungsten oxide ( $\text{WO}_3$ ) [12], and zinc oxide ( $\text{ZnO}$ ) [9]. Moreover, the authors also developed super acid catalyst for transesterification of kesambi (*Schleichera oleosa*) oil namely  $\text{ZnO}/\gamma\text{-Al}_2\text{O}_3$  (ZA) and  $\text{ZnO-CuO}/\gamma\text{-Al}_2\text{O}_3$  (ZCSA). The result of this study shows that ZCSA catalyst gives low activity in this reaction producing low yield of fatty acid methyl ester (FAME), only 35% at 65 °C of transesterification [1]. Otherwise, ZA catalyst gives better result yielding 92.2% of biodiesel at the same condition [1].

The main weakness of this acid catalyst is its active site could be reduced easily by hydration of hydroxyl acid (OH) group which is combination of Bronsted acid sites with the presence of  $\text{H}_2\text{O}$  [9,13]. Carbon based acid catalyst will be a promising heterogeneous catalyst for biodiesel production because hydration of OH group can be prevented with carbon sheet hydrophobic characteristics [13,14]. In this study, multi-walled carbon nanotubes (MWCNTs) was used as catalyst support in order to develop carbon based solid catalyst. MWCNTs was chosen as catalyst support due to its high surface area, high purity, and well developed surface morphology and porosity [14]. Furthermore, zinc oxide ( $\text{ZnO}$ ) as acid promoter was doped inside the pore of MWCNTs via Stober-like process followed by precipitation method. This catalyst ( $\text{ZnO}/\text{MWCNTs}$ ) subsequently was tested by transesterification of kesambi (*Schleichera oleosa*) oil which is has high FFA content.

## 2. Material and Methods

### 2.1. Materials

Multi-walled carbon nanotubes (MWCNTs) was supplied by Advanced Material Esoterica, China. The other analytical grade reagents with no further purification for catalyst preparation including methanol, sodium hydroxide, zinc chloride, ammonia, and ethanol was supplied by Merck, Germany. Kesambi (*Schleichera oleosa*) oil as a low grade oil (LGO) feedstock was provided from the local market. In addition, methanol with commercial grade level as transesterification reactant was purchased from the domestic chemical supplier. Then, all standard reagents for Gas Chromatography (GC) analysis were also provided by Merck, Germany.

### 2.2. Methods

#### 2.2.1. Preparation of multi-walled carbon nanotubes supported zinc oxide catalyst

Modification of Stober-like process that was proposed by Mukenga, et al. followed by precipitation method was used for the catalyst preparation [15]. This method aims to synthesize  $\text{ZnO}$  directly inside the pore of carbon nanotubes so its metal-oxide will be an acid active site on the surface of MWCNTs. Zinc methoxide would be used as precursor and was synthesized from zinc chloride ( $\text{ZnCl}_2$ ) with sodium hydroxide in alcohol solution. First, 3 grams of sodium hydroxide was mixed in 100 mL of methanol (90% v/v) to form sodium methoxide. Then, this produced sodium methoxide was reacted for 3 hours with 1 M of zinc chloride solution at room temperature. After 3 hours, zinc methoxide

solid would be formed and the solution was centrifuged for 5 minutes to separate its solids from the solution. This zinc methoxide solid, then was washed twice by methanol solution and dried at 45 °C.

Two solutions were prepared to synthesize and directly introduce ZnO into the pores of MWCNTs. This alkoxide precursor was dissolved in ethanol solvent as first solution with concentrations according to 15% wt of ZnO loading inside MWCNTs. On the other hand, the second solution was made by dispersing 10 grams of MWCNTs with 59 mL of ammonia as catalyst into 100 mL of water solvent. Then, the first solution was added slowly to the second solution and mixed for 3 hours to form ZnO nanoparticle inside the nanotubes pore. The precipitated solid was respectively separated, washed with alcohol solvent, and dried overnight at 80 °C. After drying process, the solid catalyst was crushed and calcined for 5 hours in muffle furnace at various temperatures. There are several calcination temperatures ranging from 450 – 600 °C used in this research.

The synthesized catalyst was characterized by some analysis to observe its properties. X-Ray diffraction analysis was used for investigation of crystal structure of solid catalyst. The X-ray diffraction analysis of ZnO/MWCNTs catalyst was performed by PANalytical X'Pert Pro MPD. Afterwards, N<sub>2</sub> (nitrogen) adsorption-desorption analysis followed by Brunauer-Emmett-Teller (BET) calculation method was utilized to determine total surface area of catalyst. The morphology of the catalyst surface was captured by scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM-EDX) analysis. The element composition inside the solid catalyst could be analyzed using SEM-EDX so the metal content in the catalyst would be discovered.

#### 2.2.2. Transesterification of kesambi (*Schleichera oleosa*) oil using ZnO/MWCNTs catalyst

ZnO/MWCNTs catalytic activity was tested into transesterification of kesambi (*Schleichera oleosa*) oil in a batch reactor. Kesambi oil is one of low grade oil that contains gum impurities and has high FFA content. Before esterification and transesterification was conducted simultaneously, kesambi oil must be purified to relieve its impurities called degumming process. Degumming process was carried out by adding phosphate acid (H<sub>3</sub>PO<sub>4</sub>) solution with amount of 0.1% (v/v of oil) and heating at 70 °C. After this process, the mixture was washed with aquadest to dissolve residual phosphate and left over 48 hours until three layers was formed. Oil was separated from its gum and heated at 110 °C overnight to remove residual water content.

Transesterification of kesambi oil was carried out via batch process using prepared ZnO/MWCNTs solid catalyst. A batch reactor was utilized in this experiment including three necked round bottom flask equipped with reflux condenser. The batch reactor, then was immersed in water bath and this system was heated at 65 °C of methanol boiling point. ZnO/MWCNTs catalyst with amount of 3% (w/w of oil) and 15 %wt loading of ZnO was used in this experiment. Moreover, 1:15 of oil to methanol molar ratio was also utilized as a fixed parameter condition. There are five prepared catalysts from various calcination temperatures was used in this experiment ranging from 450 – 600 °C with 50 °C of increment. These catalysts was coded by ZT4 for 400 °C, ZT4.5 for 450 °C, ZT5 for 500 °C, ZT5.5 for 550 °C, and ZT6 for 600 °C respectively.

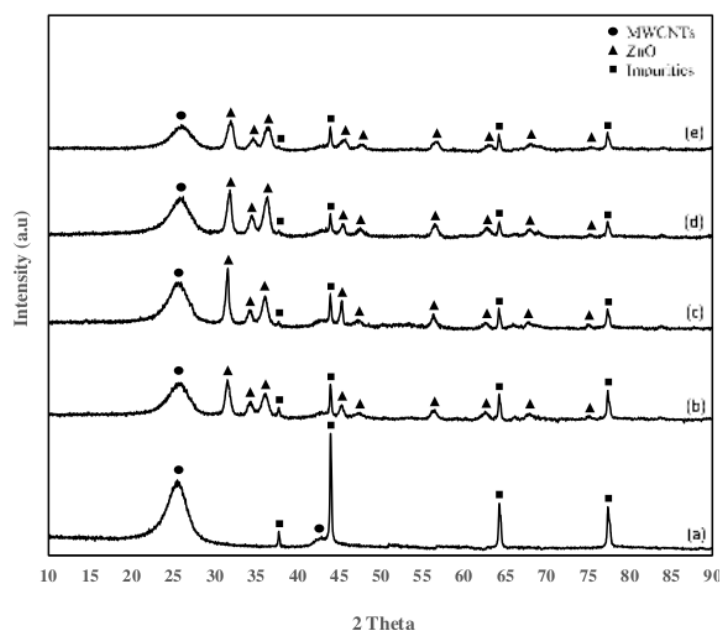
The transesterification was conducted under 150 rpm of vigorous stirring for 3 hours at methanol boiling point temperature. After the reaction was finished, the reactor was naturally cooled down to room temperature. Then, the mixture was withdrawn from the reactor and the solid catalyst was separated from the liquid via filtration process. After that, the liquid mixture was left overnight and two layers was formed that is FAME and unreaction oil in the top layer and mixture of glycerol product with excess methanol in the bottom. Finally, the FAME product was separated physically and was dried in oven to relieve the residual methanol.

Fatty acid methyl ester (FAME) content in the product was measured with a gas chromatography flame ionization detector (GC-FID) using a GC HP 5890 equipped with 20 m carbowax column. Nitrogen with a flow of 28 mL min<sup>-1</sup> was utilized as the carrier gas in this analysis. The operational conditions of analysis consist of the initial temperature and the initial time that was set by 150 °C and 3 minutes respectively. Furthermore, the increment temperature in GC equipment was set by 7.5 °C per minute until 275 °C as a final temperature was reached. The same procedure of GC analysis for biodiesel product had been used in the previous work [1]. The yield of biodiesel and the conversion of transesterification was calculated based on the amount of FAME in the product from GC analysis.

### 3. Results and Discussion

#### 3.1. Characteristics of ZnO/MWCNTs catalyst

The X-ray diffraction analysis was performed to measure the crystal structure of ZnO/MWCNTs solid catalysts. This analysis was also used in order to detect the synthesized ZnO presence inside the pore of catalyst support. Its detection was shown from diffraction peaks that similar with ZnO diffractogram pattern. The dispersed of ZnO on the carbon nanotubes surface would be an active acid sites and increase the activity of the catalyst [9]. This is 15 %wt loading of ZnO into MWCNTs that applied in this study. From figure 1, it is shown that zinc oxide as an active metal to enhance transesterification kinetic had successfully loaded into MWCNTs. The peak reference of ZnO was given by JCPDS (Joint Committee of Powder Diffraction Standard) card no. 00-036-1451. This reference has diffraction peak at  $2\theta$  angle of 31.4; 33.8; 35.8; 46.8; 55.9; 61.7; 65.5; 66.9; 68.2; 71.5; 75.8; 79.7; 88.04 degree. The same result was shown in previous study with ZnO was dopped into  $\gamma$ - $\text{Al}_2\text{O}_3$  as a catalyst support [1]. This diffractogram pattern had indexed to ZnO hexagonal wurtzile structure and seen more crystalline from its high intensity and sharper peak [16].



**Figure 1.** The X-ray diffractogram of solid catalyst comparison, (a) MWCNTs; (b) ZT4.5; (c) ZT5; (d) ZT5.5; (e) ZT6

Meanwhile, diffractogram of MWCNTs shows low and wide peak indicating the amorphous structure and less crystallinity compared to ZnO structure. XRD pattern of MWCNTs shows strong and sharp peak at  $27.06^\circ$  which indicates the graphite form and appears due to the interlayer stacking of graphene sheets [17]. Furthermore, increasing the calcination temperature leads to lower and wider peak of MWCNTs. The high temperature would change the crystal structure of MWCNTs being more amorphous and having less crystallinity. The similar phenonema also appears on the diffractogram of ZT6 that calcined at high temperature ( $600^\circ\text{C}$ ) resulting wider peaks than ZT5. It can be concluded that the high temperature would transform the catalyst structure more amorphous and reduce its cristallinity.

However, the unknown sharp peaks were seen at  $38^\circ$ ;  $44^\circ$ ;  $64^\circ$ ;  $77^\circ$  meaning some impurities substances was left inside the pores of MWCNTs. These unknown peaks have similar XRD pattern

with synthesized aluminum diffractogram pattern given by JCPDS card no. 00-04-0787 [18]. This impurities were also detected by energy dispersive x-ray spectroscopy (EDX) analysis that shown  $\text{Al}_2\text{O}_3$  impurities inside the carbon nanotubes itself and depicted in table 1. This impurities content is not significant because the MWCNTs was synthesized by chemical vapor deposition (CVD) method. Carbon nanotubes synthesis using CVD is the most efficient method producing high purity of carbon nanotubes and requiring no further purification process [19]. Other impurities like sodium oxide ( $\text{Na}_2\text{O}$ ) were left on the surface of MWCNTs after an amount of metal oxide was loaded. This materials might be entrained and formed other compound alongside the desired ZnO particle during catalyst preparation.

**Table 1.** Solid catalyst composition from EDX analysis

Compound	% mass				
	MWCNTs	ZT4.5	ZT5	ZT5.5	ZT6
C	99,4	89,43	93,45	92,41	93,7
$\text{Na}_2\text{O}$	-	2,34	-	2,36	-
$\text{Al}_2\text{O}_3$	0,6	0,44	0,49	0,51	0,3
ZnO	-	5,77	4,53	2,43	4,87
Impurities	-	2,02	1,53	2,29	1,13

Table 2 depicts the resulted surface area of catalyst that influenced by calcination temperature. The total surface area was obtained from  $\text{N}_2$  adsorption-desorption data followed by Brunauer-Emmet-Teller calculation method. Multi-walled carbon nanotubes will be very promising catalyst support due to its large surface area exceed to  $600 \text{ m}^2/\text{grams}$ . Its total surface area was larger than the other type of catalyst support like  $\gamma\text{-Al}_2\text{O}_3$ , zeolite, bentonite, and hydrotalcite [2,3,20]. The total surface area of catalyst was decreased with addition of ZnO inside the nanotubes. Otherwise, each of calcination temperature makes different surface area of catalyst with the largest area is obtained when solid catalyst was calcined at  $500 \text{ }^\circ\text{C}$ . Generally, increasing calcination temperature until its sintering point will enlarge pore volume and pore diameter due to material decomposition and impurities removal resulting in high surface area [21]. However, the surface area of catalyst decreases gradually at temperature above  $500 \text{ }^\circ\text{C}$  signifying the sintering process have occurred.

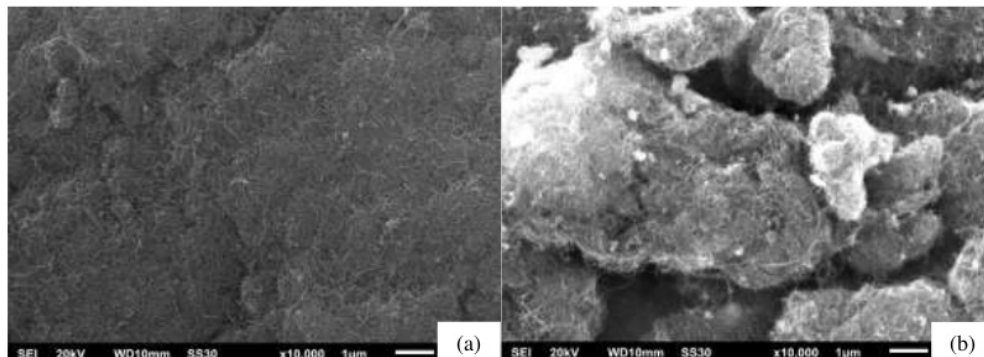
**Table 2.** Surface area of ZnO/MWCNTs catalysts with 15% ZnO loading at various calcination temperatures

Catalyst	Surface Area ( $\text{m}^2/\text{gram}$ )
MWCNTs	673,42
ZT4.5	332,111
ZT5	409,043
ZT5.5	303,062
ZT6	271,482

Moreover, the prepared solid catalysts were characterized with scanning electron microscopy (SEM) analysis. This analysis aims to capture the morphological appearance of the ZnO/MWCNTs catalyst. It is showed that MWCNTs material has randomly nanotubes arrays filling with little amount of  $\text{Al}_2\text{O}_3$  impurities (figure 2a). This morphological surface was different from other carbon nanotubes synthesizing via chemical vapor deposition (CVD) method. Usually, carbon nanotubes that prepared by CVD has typical well-alignment of nanotubes [19]. Besides that, dispersed white spotting is appeared on the entire surface of the carbon nanotubes which signifies that ZnO particle was successfully filled inside the pores of MWCNTs (figure 2b). It emphasizes the amount of ZnO and other compound that found by EDX analysis shown in table 1. Further phenomena on morphological surface of catalyst was emerged after calcination with high temperature was applied. It is shown that the catalyst became agglomerates and its surface looked broken leaving "crater-like" form broadening



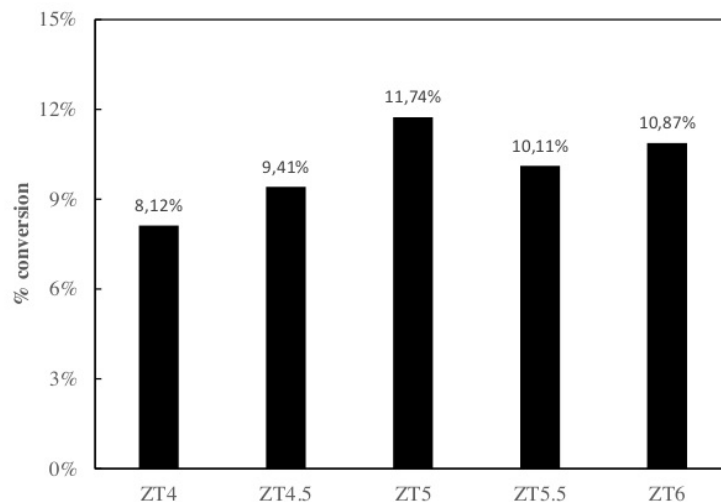
its surface area. It was reported that carbon nanotubes would be oxidated while calcined at high temperature above 400 °C releasing a number of its compound and reducing its total mass [21].



**Figure 2.** SEM image of the catalyst surface, (a) MWCNTs; (b) ZT5

### 3.2. Transesterification of kesambi (*Schleichera oleosa*) oil

The fatty acid methyl ester (FAME) as the product of reaction was measured by Gas Chromatography (GC) analysis. The activity of catalyst is specified by the active site concentration both of base and acid which in this case is zinc oxide (ZnO) loading into MWCNTs. The number of ZnO as an acid active site lead an important role in this transesterification of low grade oil. The effect of calcination temperature on this process was studied during the reaction by varying its temperature from 450 °C until 600 °C. At the same time, the other process conditions were kept constant that is 65 °C of temperature reaction, 3 hours of reaction time, and 1:15 of oil to methanol molar ratio.



**Figure 3.** Conversion of transesterification of kesambi oil using ZnO/MWCNTs catalyst

The conversion of transesterification is presented in figure 1 and generally increase while calcination temperature is raised. It is assured that transesterification would not take place without the catalyst presence and FAME might be not formed. The highest conversion of reaction is reached while ZT5 catalyst was utilized in the reaction. Then, increasing calcination temperature exceed to 500 °C properly decrease the conversion of transesterification. But, this conversion of reaction is somehow

having low value under 15% (figure 3). Contrarily, the previous study yields conversion of transesterification above 70% during addition of a few amount (1% w/w of oil) ZnO based catalyst. It might be occurred because in the previous study ZnO was dispersed over 50 %wt inside the pore of catalyst support that means there is 0,5% (w/w oil) of ZnO in the entire of oil reactant [1]. This huge amount of ZnO active acid site would increase the rate of reaction. It was different with the result of this research that is dispersing under 5 %wt of ZnO. With 3% (w/w oil) of solid catalyst in the reaction, it would be only 0,136 %wt of ZnO in the entire of process feedstock.

Theoretically, Zn<sup>2+</sup> probably has an active Lewis acid sites that gives an effect to trigger both of esterification and transesterification reaction simultaneously [9]. Because of that, ZnO would be appropriate to catalyze these reactions and could process low-grade oil that contain high FFA. But, the result was not satisfied yielding low concentration of FAME in the product. This result indicates that preparation of catalyst using modified stober-like process with impregnation and precipitation simultaneously did not give much more dispersed ZnO on the surface of the catalyst support. It was different with the previous study while used modified stober-like synthesis with sol-gel method.

#### 4. Conclusion

The multi-walled carbon nanotubes supported zinc oxide (ZnO/MWCNTs) catalyst for transesterification had been successfully prepared with the modified Stober-like process with precipitation method. Zinc oxide (ZnO) as a metal active was successfully doped inside the nanotubes of MWCNTs via this modified technique and produced solid catalyst with large total surface area. However, due to only few amount of ZnO was dispersed inside the pores of catalyst, FAME could not be produced with satisfactory results. This result encourages the authors to develop multi-walled carbon nanotubes supported zinc oxide (ZnO/MWCNTs) catalyst using other route of process in order to yield the high conversion of transesterification.

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PAGE 6

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PAGE 7

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PAGE 8

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PAGE 9

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