The effect of solvent in the hydrogenation of lauric acid to lauryl alcohol using Ru-Fe/TiO₂ catalyst

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Abstract. We describe the effect of solvent in the hydrogenation of lauric acid to lauryl alcohol using bimetallic ruthenium-iron supported on titanium oxide (5.0Ru-xFe/TiO₂ (5.0 is loading amount of Ru (wt%) and x = 1.0; 3.0; 5.0 is the loading amount of Fe (wt %). Ru-xFe/TiO₂ catalyst was synthesized via the hydrothermal method at a temperature of 150 °C for 24 hours, then followed by reduction with H₂ at a temperature of 500 °C for 3 h. The catalytic reactions were carried out in a batch reactor system in various organic solvents (e.g., 2-propanol, water, 1,4-dioxane, tetrahydrofuran, and its mixture solvent). Results of XRD analysis of 5.0Ru-xFe/TiO₂ showed a typical diffraction peak at 2θ = 44.14°, which can be attributed as metallic Ru(101). The best solvent used was 1,4-dioxane: water (4.5:0.5 ml) at 170 °C, initial H₂ pressure 4.0 MPa and a reaction time of 5 h, providing 39.4% lauric acid conversion and 5.8% yield of lauryl alcohol using 5.0Ru-1.0Fe/TiO₂ catalyst. The yield of lauryl alcohol could be enhanced up to 33% (at 49.2% conversion) after the reaction time was prolonged to 30 h under the same reaction conditions.

1. Introduction
Fatty acids and its common derivatives fatty alcohols, typically in the form of long-chain fatty alcohols such as lauryl, octyl, cetyl, and stearyl alcohols, are important intermediate in the fragrances, pharmaceuticals, detergents, emulsifiers, lubricants, cosmetics, shampoos, and conditioners industries [1]. Fatty acids contain carboxylate group, which the carbonyl group in carboxylate group has weak polarizability and has, consequently, lower reactivity than that of the common carbonyl compounds such as aldehyde and ketone. Therefore, extreme conditions are typically required where control of the selectivity of the process to the desired fatty alcohols is difficult. Bimetallic copper-chromite or zinc-chromite were the first commercial catalysts for the synthesis of fatty alcohols by hydrogenation of fatty acids under harsh temperature (e.g., up to 300 °C) and pressure (e.g., up to 30 MPa of H₂) conditions, which are potentially harmful for the environment [3]. Commercial ruthenium-based catalysts, such as Ru/C, ruthenium dioxide, Ru/γ-Al₂O₃, and bimetallic Ru-Sn/γ-Al₂O₃, has been frequently used for the acid hydrogenations under a similar high temperature and initial H₂ pressure (typically, 150-250 °C and 5-95 MPa) [2,4,19,20]. Therefore, the development of alternative catalysts for more facile and selective hydrogenation of fatty acids to fatty alcohols at lower pressures and temperatures is of great interest and is greatly challenging work.

In the past decade, there has been a substantial increase in the use oxophilic metals (e.g., Mo, W, and Y) or electropositive metals (e.g., Sn, Zn, Co, Ni) as well as the use of reducible oxide supports such as TiO₂ or MoO₃ to enhance the reactivity and selectivity of platinum group metal catalysts in the carboxylic acid hydrogenation to alcohols. For example, yttrium modified titanium oxide supported
ruthenium (Ru-Y/TiO₂) catalyst demonstrated high selectivity towards 1-hexadecanol (99%) in the hydrogenation of methyl hexadecanoate at 240 °C, 5 MPa H₂, and after 10 h [9]. Wang et al. have reported the use Ir-MoO₃/SiO₂ catalyst at 100 °C for hydrogenation of acetic acid to ethanol and initial H₂ pressure 60 bar, giving 80% conversion of acetic acid and 63% selectivity to ethanol [21]. Tomishigie and co-worker developed the combination of noble metal catalyst (M₁= Pd, Ru, Ir) with oxophilic oxides M₂O₃ (M₂= W, Y, Mo) for effective hydrogenation and hydrogenolysis reactions. The modified M₁–M₂O₃ catalysts demonstrated higher activity and selectivity than that of unmodified ones [17]. Moreover, Luo et al. reported the hydrogenation of hexadecanoic acid to alcohol using intermetallic Ru₃Snₓ/SiO₂ catalyst at 240 °C, H₂ 40 bar for 4 h, resulting in high conversion (99.0%) and selectivity to alcohol (99.7%) [11]. Huang et al. studied the supported Ru-M (M = Cu, Co, Ni) for methyl palmitate hydrogenation in n-heptane at 270 °C, H₂ 55 bar after 7 h, giving 1-hexadecanol (83%) [7], while bimetallic Ru-Zn/C catalyst only gave 7.2% conversion even after the temperature reaction increased to 300 °C [6].

In continuation of our standing interest in the liquid phase hydrogenation of biomass-derived carboxylic acids, we found that the presence of second electropositive metals (e.g. Sn) could enhance the activity and selectivity of titanium oxide-supported Ni catalyst for the hydrogenation of typical biomass-derived levulinic acid to g-valerolactone [13,14] and hydrogenation of dodecanoic acid to dodecane-1-ol [16]. In the present paper, we describe our extended studies on the hydrogenation of lauric acid into lauryl alcohol using bimetallic Ru-Fe/TiO₂ catalysts. The addition of Fe to Ru/TiO₂ catalysts improved the catalytic activity and selectivity towards lauryl alcohol. A 49.2% conversion of lauric acid and 33.0% yield of lauryl alcohol were obtained using 5.0Ru-1.0Fe/TiO₂ catalyst at 170°C, 40 bar H₂, and reaction time of 30 h in 1,4-dioxane: water (4.5:0.5 ml). The effect of reaction temperature, initial H₂ pressure, and reaction time on the yield of lauryl alcohol was also discussed systematically.

2. Materials and methods
2.1. Material
RuCl₃ (99.0%; Tokyo Chemical Industry), FeCl₃·6H₂O (99%; Merck Millipore), 2-propanol (98%, Merck Millipore), TiO₂: anatase, ethanol (96%; Merck Millipore), ethylene glycol (EG) (99.5%; Merck Millipore), NaBH₄ (95%; Tokyo Chemical Industry), NaOH (99%; Merck Millipore), H₂ gas (99.99%), N₂ gas (99.99%), lauric acid (97%; Tokyo Chemical Industry).

2.2. Catalyst preparation
A typical procedure for the synthesis of 5.0Ru-1.0Fe/TiO₂ (5.0 and 1.0 was the loading amount of Ru and Fe, respectively) described as follows [15]: RuCl₃ (0.371 mmol mmol) was dissolved in deionized water (symbolized as solution A) and FeCl₃·6H₂O (0.045 mmol) was dissolved in ethanol/ethylene glycol (2.0/1.0 volume ratio) at room temperature (symbolized as solution B). Solutions A, B, and TiO₂ (as support material; 1.0 g) at room temperature were mixed; the temperature was subsequently raised to 50°C under gentle stirring for 12 h. The pH of the mixture was adjusted to 12 through the dropwise addition of an aqueous solution of NaOH (3.1 M). The mixture was then placed into a sealed-Teflon autoclave for the hydrothermal process at 150 °C K for 24 h. The resulting black precipitate was filtered, washed with distilled water, and then dried under vacuum overnight. Prior to the catalytic reaction, the obtained black powder was treated under hydrogen at 500 °C for 3 h.

2.3. Catalyst characterization
The X-ray diffraction (XRD) analysis was performed on a Miniflex 600 Rigaku instrument with Cu as monochromatic source of CuKα radiation (λ = 0.15444 nm). The XRD was operated at 40 kV and 15 mA with a step width of 0.02°, a scan speed of 4°min⁻¹ (α₁ = 0.154057 nm, α₂ = 0.154433 nm), solar slit 1.25°, and using a Ni Kβ filter.

The BET surface area (S_BET) and pore volume (Vₚ) were measured using N₂ physisorption at -196°C on a Belsoerp Max (BEL Japan). The samples were degassed at 200 °C for 2 h to remove physisorbed gases before the measurement. The amount of nitrogen adsorbed onto the samples was
used to calculate the Brunauer–Emmett–Teller (BET) surface area via the BET equation. The pore volume was estimated to be the liquid volume of nitrogen at a relative pressure of approximately 0.995 according to the Barrett–Joyner–Halenda (BJH) approach based on desorption data [10].

The NH₃-TPD was carried out on a Belsorp Max (BEL Japan). The samples were degassed at a high temperature of 100-200 °C for 2 h to remove the analyzed gas before measurement. The temperature was then maintained at 200 °C for 2 h while rinsing with He gas. NH₃ gas (balanced NH₃, 80% and He, 20%) was introduced at 100 °C for 30 min, then evacuated with helium gas to remove the physisorbed also for 30 min. Finally, programmed temperature desorption was carried out at 100-800 °C and the absorbed NH₃ is monitored by TCD.

2.4. Catalytic reaction
Typical procedure for hydrogenation of lauric acid described as follows: Catalysts (0.05 g), lauric acid (2.0 mmol), and 2-propanol/H₂O (5 ml; 4.5: 0.5 volume ratio) as the solvent was placed into a glass reaction tube in an autoclave reactor system of TAIATSU Techno reactor (a Pyrex tube was fitted inside of a sus316 jacket to protect the vessel from corrosion in acidic media. The reactor was flushed with 0.1 MPa H₂ gas for 10 times prior to remove undesired gases. After H₂ was introduced into the reactor with an initial H₂ pressure of 4.0 MPa at room temperature, the temperature of the reactor was increased to 170 °C. After 5 h, the conversion of lauric acid and the yield of lauryl alcohol were determined by gas chromatography (GC) analysis.

2.5. Product analysis
GC analysis of the reactant (lauric acid) and products (lauryl alcohol, 1,1-dodecanediol) was performed on a Perkin Elmer Auto System XL equipped with a flame ionisation detector and Restek Rtx® BAC Plus capillary column. GC analysis was operated at detector and injector temperatures of 250 °C, N₂ as a carrier gas, rates of air and H₂ were 450 ml/min and H₂ 45 ml/min, respectively. The used 5.0Ru-1.0Fe/TiO₂ catalyst was easily separated using simple centrifugation or filtration, then finally dried overnight under vacuum at room temperature prior to reusability testing.

3. Results and discussion
3.1. Screening of solvent
In the first experiments, we evaluated the use of various solvents (e.g., 2-propanol, tetrahydrofuran (THF), 1,4-dioxane and water) as solvent media for the hydrogenation of lauric acid to lauryl alcohol using 5.0Ru-1.0Fe/TiO₂ catalyst and the results are summarised in table 1.

In alcoholic solvent, 2-propanol, 48.7% conversion of lauric acid and 4.3% yield of lauryl alcohol were obtained, whereas a relative large amount of others (e.g., iso-propyl laurate) was observed (entry 1). When 1,4-dioxane was used, the conversion of lauric acid was 41.5% with slightly higher yield of lauryl alcohol (11.4%) (entry 2), meanwhile catalytic reaction in water or in tetrahydrofuran did not occur (entries 3 and 4). This result suggests that the use of polar aprotic solvent (e.g, 1,4-dioxane) can effectively facilitate the hydrogenation. To improve the conversion of lauric acid as well as the yield of lauryl alcohol, further catalytic reactions in various mixture solvent of 1,4-dioxane were also carried out. A significant increase of laurylalcohol yield (15.8%) was obtained when 1,4-dioxane/water (4.5: 0.5 volume ratio) was used as solvent (entry 5). In 1,4-dioxane/THF and 1,4-dioxane/2-PrOH solvent mixtures, the yield of lauryl alcohol was about 10-13% (entries 6 and 7). Therefore, further evaluation was focused to 1,4-dioxane/water for next reaction to obtain high conversion and yield of the product. The amount of water to 1,4-dioxane was varied at range of 1.0, 0.5 ml, 5 drops, and 10 drops and the results are also summarized in table 1, entries 7-10. The highest yield of lauryl alcohol was 15.8% at lauric acid conversion of 39.4% in 1,4-dioxane/water (4.5: 0.5 ml). The relatively high yield of lauryl alcohol in 1,4-dioxane/water mixture solvent can be attributed to the presence of an H-bonded water molecule or single chemisorbed water on metallic surfaces that can dramatically reduce the energy span of the reaction pathway, hence enhancing the catalytic activity [12]. Alternatively, the increase of surface concentration in hydrogen atom due to dissociated water and strong interaction between water
with substrate by hydrogen bonding lowers the activation energy barrier and leads to high hydrogenation rates [5,18].

Table 1. Results of lauric acid hydrogenation over 5.0Ru-1.0Fe/TiO₂ catalyst in various solvents.

| Entry | Solvent          | Conversion (%) | Yield (%) | Lauryl alcohol | Undecane | Dodecane | Others |
|-------|------------------|----------------|-----------|----------------|----------|----------|--------|
| 1     | 2-Propanol (2-PrOH) | 48.7           | 9.3       | 0.1            | 0.0      | 17.0     |
| 2     | 1,4-Dioxane      | 41.5           | 11.4      | 1.0            | 0.0      | 13.0     |
| 3     | Water            | Trace          | Trace     | Trace          | Trace    | Trace    |
| 4     | Tetrahydrofuran (THF) | 2.1           | 1.1       | 0.0            | 0.0      | 7.2      |
| 5     | 1,4-Dioxane: THF | 29.0           | 10.7      | 0.1            | 0.1      | 0.0      |
| 6     | 1,4-Dioxane: 2-PrOH' | 41.5           | 13.9      | 0.1            | 0.0      | 7.2      |
| 7'    | 1,4-Dioxane: Water (4.5: 1.0) | 38.4           | 15.5      | 0.3            | 0.3      | 0.0      |
| 8'    | 1,4-Dioxane: Water (4.5: 0.5) | 39.4           | 15.8      | 0.2            | 0.1      | 7.0      |
| 9'    | 1,4-Dioxane: Water (4.5: 5 drops) | 34.0           | 12.8      | 0.3            | 0.2      | 0.0      |
| 10'   | 1,4-Dioxane: Water (4.5: 10 drops) | 37.8           | 12.3      | 0.0            | 0.0      | 0.0      |

Reaction conditions: lauric acid (0.4 g), catalyst (0.02 g), H₂ (4.0 MPa), solvent (5 ml); 170 °C; reaction time (5 h). aConversion and lauryl alcohol yield are determined by GC using internal standard technique. bYields of undecane, dodecane, and iso-propyl laurate were calculated by GC area ratio. cOthers are included laurate ester (e.g., iso-propyl laurate) or lauric acid dimer (e.g., lauryl laurate). dValues in the parenthesis are volume ratio of 1,4-dioxane to water (ml).

3.2. Effect of Fe loading amount

To study the insight into the role of Fe on the catalytic performance of Ru/TiO₂ catalyst in lauric acid hydrogenation, the amount of Fe-loaded was varied at range of 1.0, 3.0, and 5.0 wt% to the amount of Ru and the results are summarized in table 2. A reference catalyst of 5.0Ru/TiO₂ synthesized and reduced with H₂ at 500 °C for 3 h and showed low conversion of lauric acid (9.8%) as well as the low yield of lauryl alcohol (6.0%) (entry 1). The as-prepared 5.0Ru-1.0Fe/TiO₂ (the catalyst without reduction with H₂) was completely inert for the hydrogenation of lauric acid (entry 2), suggesting the transformation from Ru oxide to metallic Ru(0) was important to obtain the active species in 5.0Ru-(x)Fe/TiO₂ catalyst. Although the conversion of lauric acid increased as the Fe loading amount was increased, the yield of lauryl alcohol was unchanged, whereas the yield of others increased gradually. Therefore, the best results for the amount of Fe co-modifier was 1.0 wt% to Ru as indicated in table 2, entry 3.

Table 2. Results of lauric acid hydrogenation over 5.0Ru-(x)Fe/TiO₂ catalysts with different loading amount of Fe.

| Entry | Catalyst          | Conversion (%) | Yield (%) | Lauryl alcohol | Undecane | Dodecane | Others |
|-------|------------------|----------------|-----------|----------------|----------|----------|--------|
| 1     | 5.0Ru/TiO₂       | 9.8            | 6.0       | 1.0            | 0.0      | 2.0      |
| 2'    | 5.0Ru-1.0Fe/TiO₂ | 9.3            | 0.0       | 0.0            | 0.0      | 9.0      |
| 3     | 5.0Ru-1.0Fe/TiO₂ | 39.4           | 15.8      | 0.2            | 0.1      | 7.0      |
| 4     | 5.0Ru-3.0Fe/TiO₂ | 58.7           | 14.4      | 0.1            | 0.0      | 12.1     |
| 5     | 5.0Ru-5.0Fe/TiO₂ | 53.1           | 13.0      | 0.0            | 0.0      | 16.5     |

Reaction conditions: lauric acid (0.4 g), catalyst (0.02 g), H₂ (4.0 MPa), solvent 1,4-dioxane: water (4.5:0.5 ml); 170 °C; reaction time (5 h). aConversion and lauryl alcohol yield are determined by GC using internal standard technique. bYields of undecane, dodecane, and iso-propyl laurate were calculated by GC area ratio.cOthers are included laurate ester (e.g., iso-propyl laurate) or lauric acid dimer (e.g., lauryl laurate). dThe catalyst without reduction with H₂ (as-prepared).
3.3. Effect of initial H2 pressure

The effect of initial H2 pressure on the conversion and yield in the lauric acid hydrogenation was evaluated and the results are summarised in table 3. A 36.9% conversion of lauric acid and 2.8% yield of lauryl alcohol were obtained at initial H2 pressure of 2.0 MPa (entry 1). However, a high yield of others (included laurate ester (e.g., iso-propyl laurate) or lauric acid dimer (e.g., lauryl laurate)) was also obtained, suggesting the main reaction was not hydrogenation owing to lower H2 presence. Interestingly, the yield of lauryl alcohol remarkably increased to 15.8% at 39.4% conversion of lauric acid when the initial H2 pressure was increased to 4.0 MPa (entry 2). The yield of others also significantly decreased to 7.0%.

Table 3. Results of lauric acid hydrogenation over 5.0Ru-1.0Fe/TiO2 catalyst at different initial H2 pressure.

| Entry | Initial H2 pressure (MPa) | Conversiona (%) | Yielda (%) | Undecaneb | Dodecaneb | Othersc |
|-------|---------------------------|-----------------|------------|-----------|-----------|---------|
| 1     | 2.0                       | 36.9            | 2.8        | 0.1       | 0.1       | 19.0    |
| 2     | 4.0                       | 39.4            | 15.8       | 0.2       | 0.1       | 7.0     |

Reaction conditions: lauric acid (0.4 g), catalyst (0.02 g), solvent 1,4-dioxane: water (4.5:0.5 ml); 170 °C; reaction time (5 h). Conversion and lauryl alcohol yield are determined by GC using internal standard technique. aYields of undecane, dodecane, and iso-propyl laurate were calculated by GC area ratio. bOthers are included laurate ester (e.g., iso-propyl laurate) or lauric acid dimer (e.g., lauryl laurate).

3.4. Kinetic profiles

The kinetic profiles of lauric acid hydrogenation to lauryl alcohol in the presence of 5.0Ru-1.0Fe/TiO2 catalyst are summarised in table 4. At an early reaction time of 2 h, the main product was others (included laurate ester (e.g., iso-propyl laurate) or lauric acid dimer (e.g., lauryl laurate)) (6.9%) while the yield of lauryl alcohol was only 3.3% (entry 1). A remarkable increase of lauryl alcohol yield (15.8%) was obtained after a reaction time of 5 h at 39.4% conversion (entry 2). The yield of lauryl alcohol gradually increased as the reaction time was prolonged up to 30 h, whereas the highest yield of lauryl alcohol (33.4%) was obtained at 49.2% conversion of lauric acid (entry 5). These results suggest that the slow activation of lauric acid by catalyst is the main factor for the hydrogenation reaction to corresponding lauryl alcohol as well as the use of appropriate solvent as mentioned above. Alternatively, the applied reaction temperature higher than 170 °C may be enhanced the conversion and yield of targetted product. The catalytic reactions under temperature of 200 °C using the same catalyst were being investigated and will be described in our succeeding paper.

Table 4. Kinetic profiles of lauric acid hydrogenation in the presence of 5.0Ru-1.0Fe/TiO2 catalyst.

| Entry | Reaction time (h) | Conversiona (%) | Yielda (%) | Undekanac | Dodekane | Othersd |
|-------|-------------------|-----------------|------------|-----------|----------|---------|
| 1     | 2                 | 11.3            | 3.3        | 0.0       | 0.0      | 6.9     |
| 2     | 5                 | 39.4            | 15.8       | 0.2       | 0.1      | 7.0     |
| 3     | 15                | 41.9            | 27.2       | 0.4       | 0.1      | 5.3     |
| 4     | 24                | 42.8            | 30.1       | 1.1       | 1.8      | 5.5     |
| 5     | 30                | 49.2            | 33.4       | 3.7       | 2.5      | 4.7     |

Reaction conditions: lauric acid (0.4 g), catalyst (0.02 g), solvent 1,4-dioxane: water (4.5:0.5 ml); 170 °C; reaction time (2-30 h). aConversion and lauryl alcohol yield are determined by GC using internal standard technique. bYields of undecane, dodecane, and iso-propyl laurate were calculated by GC area ratio. cOthers are included laurate ester (e.g., iso-propyl laurate) or lauric acid dimer (e.g., lauryl laurate).
3.5. Structure-activity relationship

On the basis of spectrometric characterization results, surface acidity, and reaction profiles, we discuss the structure of active Ru and the presence of FeO_x species in Ru-xFeO_x/TiO_2 catalyst after reduction with H_2 at 500 °C for 3 h. The as-prepared 5Ru-1.0FeO_x/TiO_2 was a referenced catalyst without reduction with H_2 and inactive for hydrogenation of lauric acid (table 1, entry 2). The XRD patterns of Ru-xFeO_x/TiO_2 catalysts before and after reduction with H_2 at 500 °C for 3 h (figures 1 (a) and (b)) suggested that the formation of metallic Ru(0) was observed at 2θ = 44.34° corresponding to Ru(101) (21). On the other hand, the metallic or oxide Fe species were unable to detect due to its crystallite sizes lower than that the detection limit of XRD.

![Figure 1](image1.png)

**Figure 1.** XRD patterns of (a) before and (b) after reduction with H_2 at 500 °C for 3 h with different amount of Fe; (a) 5.0Ru-1.0Fe/TiO_2, (b) 5.0Ru-3.0Fe/TiO_2, and (c) 5.0Ru-5.0Fe/TiO_2.

The interaction between active surface of catalyst and lauric acid substrate and lauryl alcohol product was also monitored by ex-situ ATR-FTIR analysis and the results are shown in figure 2. A specific absorbance of -OH was observed on recovered-5.0Ru-1.0Fe/TiO_2 catalyst (figure 2f), the absorbance of -OH totally disappeared after the recovered-5.0Ru-1.0Fe/TiO_2 catalyst was dried at room temperature under vacuum for 7 h (figure 2g).

Interestingly, a sharp absorbance peak was observed at a wave number of 1728.1 cm⁻¹ which can be attributed as the finger print of carboxylic acids suggesting the lauric acid absorbed strongly on catalyst surface and the absorbed carboxylic acid remained even after dried under vacuum at room temperature (figures 2f and 2g). In addition, the results also suggest to remove all absorbed molecule on the catalyst surface that may affect to the quantify both remained reactant (lauric acid) and the formed product (lauryl alcohol) using GC analysis and finally may deviate the number of conversion as well as the yield. Therefore, it is suggested to dilute the reaction mixture by addition of appropriate solvent before GC analysis and it is being investigated in our research group.
Figure 2. ATR-FTIR spectra of (a) dodecane, (b) lauric acid, (c) lauryl alcohol, (d) 1,4-dioxane, (e) fresh 5.0Ru-1.0Fe/TiO$_2$ catalyst, (f) recovered 5.0Ru-1.0Fe/TiO$_2$ catalyst, (g) recovered 5.0Ru-1.0Fe/TiO$_2$ catalyst, dried at room temperature under vacuum.

4. Conclusions
We described the hydrogenation of lauric acid to lauryl alcohol in the presence of 5.0Ru–(x)Fe/TiO$_2$ catalysts. The best solvent used was 1,4-dioxane: water (4.5:0.5 ml) at 170 °C, initial H$_2$ pressure 4.0 MPa and a reaction time of 5 h, providing 39.4% lauric acid conversion and 5.8% yield of lauryl alcohol using 5.0Ru–1.0Fe/TiO$_2$ catalyst. The yield of lauryl alcohol could be enhanced up to 33% (at 49.2% conversion) after the reaction time was prolonged to 30 h under the same reaction conditions.

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