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Catalytic dehydration of 2-propanol over nickel phosphide immobilized on natural bentonite

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Abstract

This study investigated the fabrication of nickel phosphide immobilized on natural bentonite (NiP/bentonite) as a low-cost catalyst with different metal loadings (mEq/g) to produce diisopropyl ether (DIPE) as a potential gasoline additive. The catalysts were prepared using a simple wet-impregnation method. The physico-chemical features of the parent natural bentonite and NiP/bentonite were analyzed using FTIR, SEM–EDX, XRD, N₂ physisorption, and catalyst acidity using the NH₃ gravimetric method. The dehydration reaction was conducted under mild conditions (150 °C for 3 h) using a reflux system. The results suggested that the NiP species did not alter the bentonite structure while increasing its acidity and surface area properties. Using the 8 mEq/g NiP/bentonite catalyst, a DIPE product of 68.52% was achieved owing to the highly acidic sites of the catalyst and high stability over three cycles. This study highlights the potential of nickel-phosphide-supported natural bentonite in the dehydration reaction for DIPE production.

Keywords Diisopropyl ether · 2-propanol dehydration · Nickel phosphide · Natural bentonite

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Introduction

Natural bentonite is the most frequently discovered mineral in Asia, particularly in Indonesia. This mineral is a silicate crystal with a layered structure [1]. Natural bentonite can expand and has a high ion exchange capacity, allowing it to absorb large amounts of interlayer cations [2]. This material has been extensively employed in many applications such as biolubricants [3], dye removal [4], hydrocracking catalysts [5, 6], phosphate removal [7], and Cr removal [8]. Under these conditions, the workability of bentonite is insufficient [9]. Because the properties of natural bentonite can be tuned, it is imperative to make modifications to enhance its performance and thus obtain a better product that inherently promotes its utilization for many potential applications [10]. The bentonite modification technique aims to improve the physicochemical features. The development of bentonite-based materials as catalysts and supported catalysts has proliferated because they are inexpensive and readily abundant [11]. In the context of the catalyst, textural and acidity features crucially affect the catalytic activity. One fabrication approach can be established by dispersing or incorporating the active species into the pore and/or the surface bentonite [12].

Presently, various species such as Fe, Ti, Zr, Al [12], Sn, Cr, LDH-based on polyoxo-vanadate and molybdate [13], Al/Fe [14], Zr [15], Co [16], Zr–Al [11], and Fe–Cr [17], have been explored and combined with bentonite. Recently, transition metal phosphide-based catalysts have been explored because of their high catalytic activity and selectivity for hydrogen and oxygen evolution reactions [18]. These materials are phosphorus transition metal compounds that combine the physical features of ceramics, including hardness and strength, with the electrical properties of metals, such as conductivity. Transition metal phosphides have been proposed as favorable catalysts owing to their low cost, abundance, and high efficiency [19]. The metal and P sites on the catalyst surface behave as proton acceptors and hydride receptor centers and are capable of replacing noble metal catalysts such as Pt [20]. The presence of phosphorus, which exerts a “ligand” (or electronic) effect and is geometrically situated at the location of the metal, has been attributed to the outstanding performance of transition metal phosphides [21]. A series of Ni₂P, WP, Fe₂P, CoP, and MoP have been fabricated and employed for the hydrodesulfurization of fuel [22]. Metal phosphide-based catalysts have also been utilized in hydrodeoxygenation reactions [19–21, 23–26]. Among the transition metal phosphide catalysts, nickel phosphide catalysts have been extensively reported to have high surface areas and high catalytic activities for many applications, such as HDS, dehydrogenation, hydrodeoxygenation, electrocatalytic water splitting, and methane dry reforming [27–29]. de Souza et al. [20] have developed the NiP species supported on SiO₂, Al₂O₃, CeO₂, TiO₂, and CeZrO₂. Ruangudomsakul et al. [26] reported that the Ni_xP_y catalyst provided a high green diesel yield in the dehydrogenation of palm oil compared with CoP and CuP. Gonçalves et al. [21] revealed that the Ni₂P-supported ZrO₂ has higher HDO activity than Ni₂P/SiO₂. Wagner et al. [30] developed NiP-supported zeolite Y for quinoline hydrodenitrogenation.

To the best of our knowledge, no research has been done on the preparation of nickel phosphide-supported natural bentonite. The combination of these species provides a positive effect with a highly acidic active site and surface area, as well as a low-cost catalyst. Therefore, in the present study, a nickel phosphide-supported catalyst was used as a model for the dehydration of 2-propanol to diisopropyl ether. Diisopropyl ether (DIPE), as reported in the literature, has the potential to be used as a gasoline additive with the potential to reduce GHG emissions and other GHG emissions [31]. DIPE has good features that can increase the octane number of gasoline fuel and is environmentally friendly [32], compared with other chemicals such as MTBE, ETBE, and TAME [33]. In this regard, various catalysts have been utilized for 2-propanol dehydration [34–39], but nickel phosphide-supported natural bentonite has not been comprehensively discussed. The DIPE product was evaluated using nickel phosphide-supported bentonite and parent natural bentonite. The physicochemical features of the catalyst will be assessed using a series of analyses, such as XRD, FTIR, SEM–EDS, N_2 adsorption–desorption, and total acidity by the gravimetric method. The dehydration product will be investigated using GC–MS.

Experimental

Preparation of nickel phosphide immobilized on bentonite catalysts

In a typical preparation, natural bentonite (200-mesh) from Bayan was first saturated with NaCl solution ($\geq 99.5\%$ purity, Merck), as reported in a previous study [9]. The as-prepared Na-bentonite (5 g) was subsequently dispersed in 0.1 M $NiCl_2 \cdot 6H_2O$ solution ($\geq 98.0\%$ purity, Merck) with various volumes and stirred for 1 h at room temperature. Afterward, 0.5 M $(NH_4)_2HPO_4$ solution ($\geq 99.0\%$ purity, Merck) was gradually dripped onto the solution until it reached a predetermined volume corresponding to nickel phosphate loadings of 2, 4, 6, and 8 mEq/g of catalyst. The solution was stirred at room temperature and the temperature was gradually increased until a paste was formed. The paste was dried at 80 °C for 1 day and calcined at 350 °C for 30 min. The as-synthesized nickel phosphate-bentonite (~5 g) was dissolved in 50 mL of DW and reduced using a certain amount of $NaBH_4$ powder ($\geq 96.0\%$, Merck) under a sonicator for 30 min. The powder was separated and dried at 80 °C for 1 day. The 200-mesh dried nickel phosphide-supported bentonite is denoted as NiP/bentonite.

Characterization of catalyst

The phases and crystal structures of Na-bentonite and NiP/bentonite were characterized using a Rigaku Mini Flex 600 Powder Diffractometer. The 2θ range of measurements from 5° to 80° with a step size of 0.0200° and a count time of 0.24 s. Shimadzu FTIR 8201 (KBr pellet method) was used to analyze the functional groups of both catalysts, with range of measurements from 4000 to 450 cm^{-1} . SEM micrographs of the catalysts were obtained using an SEM JSM 650, and the

elemental composition was determined using EDS. The textural properties of the catalysts were evaluated using Quantachrome NOVA. Prior to analysis, the catalyst was vacuum degassed up to 300 °C for 1 h with a heating rate of 10 °C/min. The surface areas of the catalysts were calculated using a multipoint BET procedure. The total acidity of the catalysts was determined gravimetrically using NH₃ as the probe. Before adsorption, the desiccator was vacuumed, saturated, and flowed with NH₃ vapor from the NH₄OH solution for 1 day (≥25.0% purity, Merck). The as-adsorbed NH₃ catalyst samples were quantitatively weighed, and the NH₃ absorbed catalyst (mmol NH₃/g catalyst) was calculated according to a previous study [9].

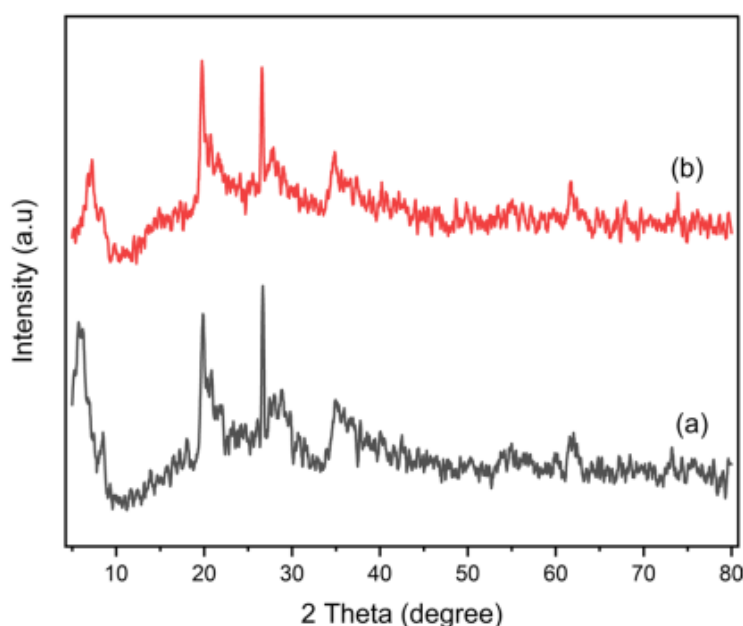
Catalytic activity test

In the catalytic activity test, 50 mL of 2-propanol (≥99.8% purity, Merck) was introduced into the boiling flask, followed by 0.5 g catalysts under the batch reactor. The dehydration was conducted at 150 °C for 210 min. The catalyst was then separated, and the product was analyzed using a Thermo Scientific GC–MS instrument. To investigate the reusability of the catalyst, the spent catalyst in the first cycle was washed several times with acetone, dried overnight at 120 °C, and used for the next cycle.

Results and discussion

Physicochemical characterization

The crystal structure and phases of the as-synthesized Na-bentonite and NiP/bentonite were assessed using XRD, as shown in Fig. 1. As shown in Fig. 1a, a

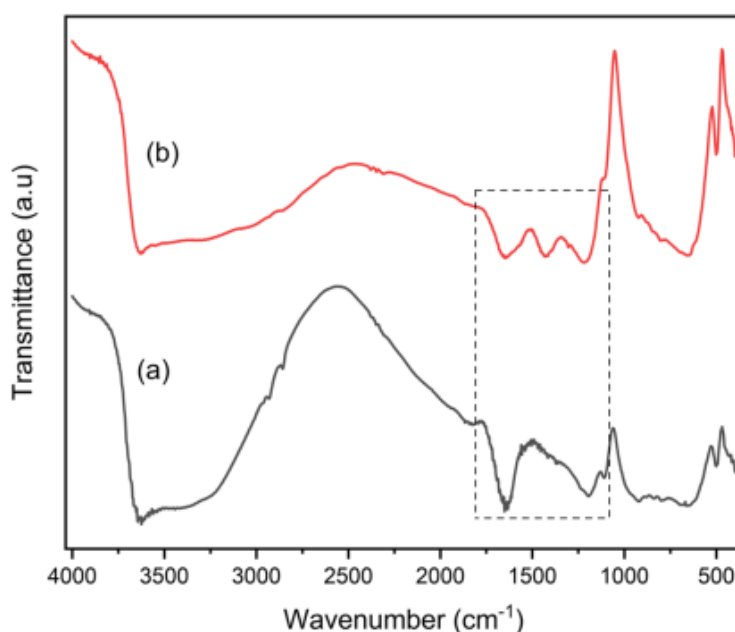


37 Fig. 1 XRD pattern (a) Na-bentonite and (b) NiP/bentonite

characteristic of the montmorillonite phase was observed at $2\theta=6.78^\circ$, 21.21° , 35.23° , and 62° [40]. Quartz phase impurities were also observed at $2\theta=26.43^\circ$ and 29.23° [41]. These typical bentonite peaks have been consistently reported in other studies [42–44]. A typical nickel phosphide phase, as in other reports [22, 26, 45], is likely unobserved in the NiP/bentonite diffractogram (Fig. 1b), which suggests that this phase is amorphous [20]. This condition was also reported by González-castaño et al. [27], who reported that a highly crystalline phase of nickel phosphide could be achieved during high-temperature calcination. No appreciable change in the diffractogram of Na-bentonite after loading with nickel phosphide indicated that the main structure of bentonite was favorably maintained [21].

The IR bands characteristic of functional groups of parent Na-bentonite and NiP/bentonite are shown in Fig. 2. The absorption band at 3582 cm^{-1} (Fig. 2a) was related to the stretching vibration of the metal-OH in the octahedral layer of bentonite [46], whereas the adsorbed H groups or water molecules can be found at 1625 cm^{-1} [4, 47]. The absorption band at 1035 cm^{-1} was assigned to the stretching vibration of the Si–O–Si bond and the absorption band at 922 cm^{-1} was attributed to the Al–OH vibration [48]. Al, Si–O, and Al–O–Si coexisted vibrations were observed at ca. 692 cm^{-1} , 432 cm^{-1} , and 761 cm^{-1} , respectively [49]. The FTIR spectrum of NiP/bentonite exhibited a distinct absorption band at 1350 cm^{-1} , which was attributed to the vibration of phosphate groups from NiP species [50–53]. NiP/bentonite displayed absorption bands similar to those of the parent bentonite, which is consistent with the XRD results.

SEM micrographs of both catalysts are presented in Fig. 3. Na-bentonite has sizeable aggregations of irregularly distributed platelets [54]. These platelets have a lamellar structure with sharp edges [55, 56]. No appreciable surface morphological changes were observed in the SEM micrograph of NiP/bentonite (Fig. 3b).



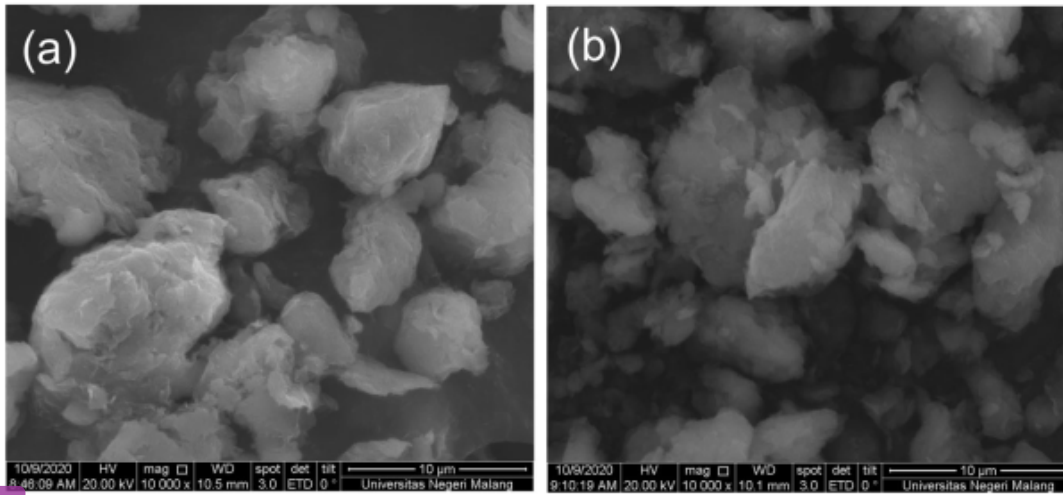


Fig. 3 SEM micrographs **a** Na-bentonite and **b** NiP/bentonite

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A similar finding was reported by Pajarito et al. [48], who revealed no significant change in the surface morphology of Zn-bentonite compared with that of the parent bentonite.

EDS analysis (Table 1) showed that the Si/Al ratio was relatively unchanged, which suggested that the NiP species loaded towards the bentonite surface did not alter the main frameworks of bentonite [5], which is consistent with the XRD and FTIR results. Furthermore, P (1.66 wt.%) and Ni (3.23 wt.%) elements co-existed in the NiP/bentonite, which suggested that the NiP/bentonite catalyst was successfully prepared. Table 1 shows that Na-bentonite consists of various elements, as has been consistently reported in other studies [54]. The pre-existing P elements before Na-bentonite modification were possible because they originated from nature, although the content of the P element increased after modification. Abdou et al. [57] reported a similar finding when preparing Egyptian bentonite

Table 1 Elemental analysis of catalysts using EDS

Element	Atomic (wt.%)	
	Na-bentonite	NiP/bentonite
C	14.42	24.25
O	47.25	42.82
Na	2.02	2.25
Mg	2.42	2.08
Al	8.4	5.62
Si	20.8	13.92
Ca	0.45	0.37
Fe	3.51	2.78
Cl	0.61	0.73
P	0.12	1.66
Ni	0	3.23

($P_2O_5 = 0.16\%$). The EDS results suggest that the NiP species were capable of supporting bentonite without changing its main framework.

The N_2 adsorption–desorption curves of all catalysts are shown in Fig. 4. It can be seen that all catalysts had a type IV isotherm based on IUPAC categorization, which mainly indicated that the catalysts were mesoporous [58, 59]. de Oliveira et al. [55] stated that the disjunction of the adsorption–desorption curve at a relative pressure (ca. 0.4) is attributed to the appearance of small mesopores on the adsorbate. Monolayer nitrogen adsorption on the mesoporous wall occurred when P/P_0 was below 0.1 and capillary condensation occurred in mesopores with P/P_0 in the range of 0.4–0.8, which was revealed by a sharp increase and hysteresis in the adsorption isotherm. Multilayer adsorption can be observed on the outer surface of the particles at higher P/P_0 [58]. The hysteresis in Fig. 4 is recognized as type H4, which suggests a slit-shaped porous solid [14]. A previous study reported similar observations for other clay-based materials [3].

The textural features of the catalysts are presented in Table 2. It can be seen that the as-prepared NiP/bentonite surface area, diameter, and volume of pores were much higher than those of Na-bentonite. The surface area of catalysts increased as the metal loading increased. The increase in the corresponding textural features suggests that impregnation with NiP species promoted an increase in porosity and surface area. The increase in the BET surface areas of pillared natural bentonite samples may be attributed to the increase in pore volume and enlarged total pore volume owing to the presence of NiP species [60]. Another study also reported the same trend [61, 62]. Compared with a previous study [9], NiP/bentonite had a higher surface area than MoP-bentonite ($63.69 \text{ m}^2/\text{g}$), which promoted more reactant to be adsorbed on the surface and affected the dehydration process over the catalyst's active site. However, prolonged metal loading (10 mEq/g) decreased the surface area of the catalyst as well as the pore diameter,

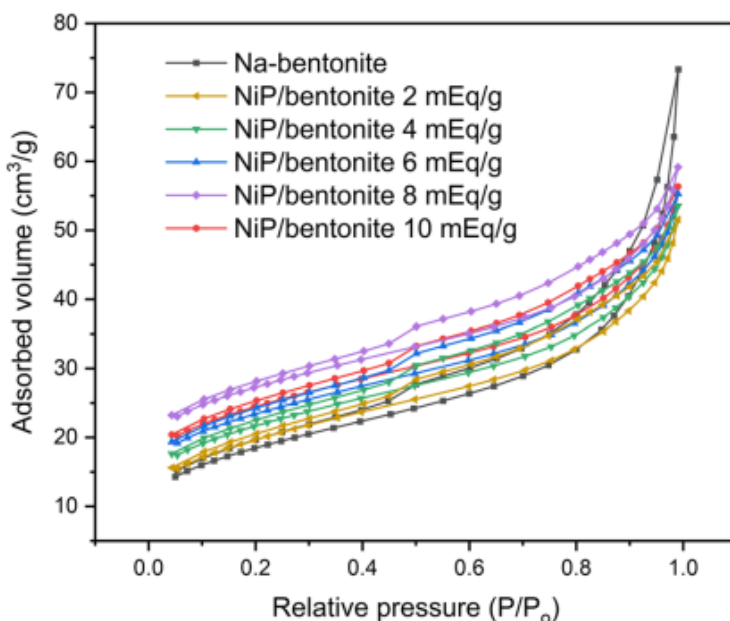


Fig. 4 N_2 adsorption–desorption isotherms of (a) Na-bentonite and (b) NiP/bentonite

Table 2 Textural features of Na-bentonite and NiP/bentonite

Catalyst	Surface area (m ² /g)	Pore diameter (Å)	Pore volume (cm ³ /g)
Na-bentonite	51	52.31	0.063
NiP/bentonite 2 mEq/g	55	54.12	0.075
NiP/bentonite 4 mEq/g	56	54.64	0.094
NiP/bentonite 6 mEq/g	61	60.24	0.079
NiP/bentonite 8 mEq/g	67	66.68	0.113
NiP/bentonite 10 mEq/g	65	64.21	0.102

suggesting the pore blockage by the NiP species. According to Table 4, catalyst with NiP metal loading of 8 mEq/g exhibited the most optimum textural features that provided the catalytic reaction of 2-propanol conversion towards DIPE product.

Table 3 presents the total acidity of Na-bentonite and NiP/bentonite with varying NiP loadings. This approach measures the weight of the catalyst before adsorption and post-adsorption of NH₃ vapor, where the amount of NH₃ gas absorbed by the catalyst is proportional to the number of acid sites on the catalyst [63]. As shown in Table 3, Na-bentonite has a total acidity of 1.45 mmol NH₃/g, which is lower than that of the corresponding NiP/bentonite catalyst. The aluminosilicate structure of the parent bentonite is primarily responsible for its low overall acidity [6]. The total acidity of Na-bentonite gradually increased as the NiP loading increased, presumably due to the availability of Lewis acid sites from Ni and Brønsted acid sites from the phosphor species. The higher the Lewis and Brønsted sites, the higher the total acidity of the catalyst [64, 65]. The highest total acidity (4.63 mmol NH₃/g) was attained by NiP loading of 8 mEq/g. Further NiP loading (10 mEq) decreased the total acidity, presumably due to agglomeration which reduced the active sites of the catalyst [66]. Wijaya et al. [67] stated that the number and distribution of active site groups on the surface that reached the maximum value caused the adsorbed base probe molecule to decrease.

Table 3 Acidity features of Na-bentonite and NiP/bentonite

Catalyst	Total acidity (mmol/g)
Na-bentonite	1.45
NiP-bentonite 2 mEq/g	2.78
NiP-bentonite 4 mEq/g	3.15
NiP-bentonite 6 mEq/g	3.57
NiP-bentonite 8 mEq/g	4.63
NiP-bentonite 10 mEq/g	3.63

Catalytic activity test

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Na-bentonite and NiP/bentonite were tested for their catalytic activity in the dehydration of 2-propanol to DIPE using a fixed-bed reactor. The dehydration reaction is appreciably contingent on metal properties and metal loading [68]. Fig. 5 shows the effect of Na-bentonite and NiP/bentonite with various loadings on the DIPE product. The low acidic sites of Na-bentonite could not provide sufficient catalytic activity for DIPE production. In comparison, when the NiP species were loaded into bentonite, the DIPE product reached 64.43% at 2 mEq/g NiP loading. Prolonged NiP loading directly increased the DIPE product to 68.52%, achieved by 8 mEq/g NiP loading, presumably due to the presence of a highly acidic active site of the catalyst [36]. Meanwhile, a high NiP loading (10 mEq/g) decreased the DIPE product to 65.22%. These results were consistent with the total acidity feature acidity, which suggests that the DIPE product was mainly accelerated by the acidity of the catalyst [69]. The NiP/bentonite catalyst showed a higher DIPE product than MoP-bentonite (64.5%) [9].

The stability performance of NiP-bentonite (8 mEq/g) on the DIPE product was evaluated after three reaction cycles (Table 4). The spent catalyst was regenerated by washing and (45) ng. The DIPE product was relatively stable within three cycles, presumably due to the strong interaction between the NiP active sites on the bentonite. However, prolonged regeneration of the catalyst probably (2) causes the loss of active sites because of the washing process, thereby decreasing the performance of the catalyst [70].

The acidity value of the reused catalyst was further evaluated to confirm the relation between the acidity value of the catalyst with the decreased catalytic activity after three consecutive runs. The first reused catalyst decreased catalyst acidity

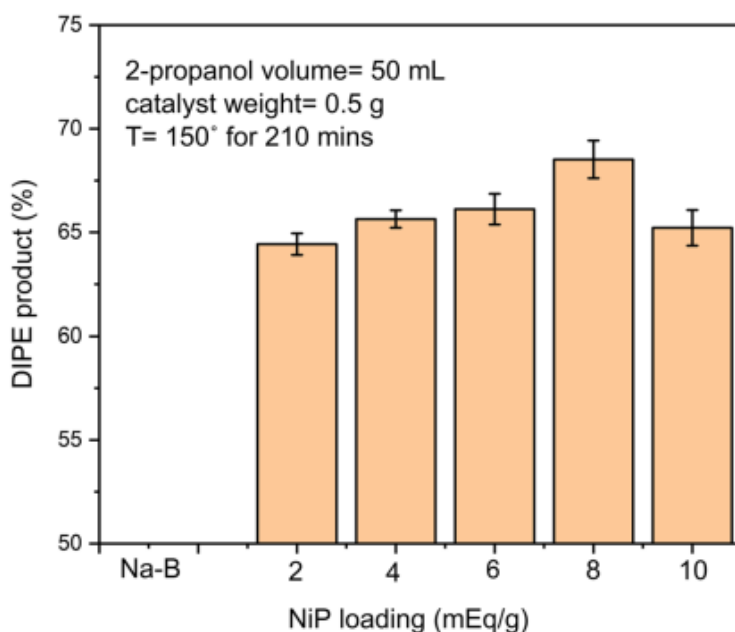


Fig. 5 DIPE production using Na-bentonite and NiP/bentonite with various NiP loading

Table 4 Reusability performance of NiP-bentonite (8 mEq/g) catalyst towards DIPE product

Cycles	DIPE product (%)
Fresh	68.50 ± 0.16
1	68.05 ± 0.11
2	67.51 ± 0.17
3	66.21 ± 0.10

from 4.63 mmol/g (fresh catalyst) to 4.59 mmol/g and tended to decrease up to 4.56 mmol/g after two consecutive runs. Moreover, the catalyst at three times reused showed a decrease in the acidity value to 4.49 mmol/g. The regeneration process could decrease catalyst acidity, implying less catalyst active site. Consequently, the catalyst performance also decreased.

Conclusion

In summary, this study evaluated the potential of nickel-phosphide-supported bentonite prepared using the wet impregnation method as an acidic catalyst for DIPE production through 2-propanol dehydration. The NiP species significantly increased the total acidity and surface area of Na-bentonite, which promoted the dehydration of 2-propanol to DIPE. XRD, FTIR, and SEM–EDX analyses revealed that the loading of NiP onto bentonite did not alter the main framework of the bentonite. A 2-propanol dehydration study showed that the NiP loading on bentonite affected the DIPE yield. The highest DIPE product, up to 68.52%, was achieved with NiP-bentonite at 8 mEq/g. This study provided a potential application of metal phosphide-supported natural bentonite for the dehydration reaction.

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Data availability All experimental data in this study is available, and we can show it at any time if anyone wants further confirmation of the data.

Declarations

Conflict of interest The authors have no competing interests to declare that are relevant to the content of this article.

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