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Abstract: Adsorption properties of different adsorbents such as reduced NiY, AgY, alumina, 13X, and activated carbon were studied with dibenzothiophene (DBT) and naphthalene as model compounds. The desorption of DBT was carried on thermo gravimetric–differential thermal analysis (TG-DTA). The interaction of DBT with different adsorbents follows the sequence: activated carbon > reduced NiY > AgY > activated alumina > 13X. The bio-regeneration of these adsorbents was studied with *P. delafieldii* R-8 as desulfurization strains. Adding *P. delafieldii* R-8 cells can improve DBT desorption from adsorbent AgY. The desorption of DBT from adsorbents by bio-regeneration of adsorbents follows the sequence: 13X > alumina > AgY > reduced NiY > activated carbon. The presence of naphthalene can decrease the desorption of sulfur compounds. The adsorption capacity of AgY decreases for the first time recycling and then changes little. The decrease of the adsorption capacity is due to the loss of Ag^{+} ions.

Keywords: Desulfurization, adsorption, *P. delafieldii* R-8, bio-regeneration, dibenzothiophene

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INTRODUCTION

Vast amounts of sulfur oxides released into the atmosphere by combustion of fossil fuels are the a principal source of acid rain. Ultra-deep removal of sulfur from transportation fuels has become very imperative for the petroleum refining industry due to the increasing stringent environmental regulations. Hydrodesulfurization (HDS) is a conventional method to remove sulfur compounds for industrial purposes. The main drawbacks of HDS include high temperature ($>300^{\circ}$C), high pressure (>4 MPa), high energy cost, and difficulty to remove aromatic heterocyclic sulfur compounds such as dibenzothiophene (DBT) and substituted DBTs completely and produce clean fuels (1). In order to achieve the "no sulfur" specification, some new technologies such as adsorption desulfurization (2–4), biodesulfurization (5), extraction with ionic liquids (6–8), and complex formation (9, 10) were proposed to remove sulfur compounds from fuels.

Desulfurization by adsorption is a method to remove sulfur compounds with modified metal oxides, molecular sieves, and activated carbon as adsorbents under ambient conditions. During the last decades there have been enormous amounts of research about adsorption desulfurization. The sulfur compounds can be removed from commercial fuels either via π-complexation (3, 11–15), van der Waals, and electrostatic interactions (16), or reactive adsorption by chemisorption (9, 10). It is reported that π complexation adsorbents and reduced NiY have higher selectivity towards sulfur compounds over aromatics (3, 17, 18, 22–24). There are large amounts of aromatics but trace amounts of sulfur compounds in diesel. Therefore, aromatics can also be adsorbed on the desulfurization adsorbents during the process of desulfurization. Solvent extraction and oxidation in the air are two methods to regenerate the desulfurization adsorbents. But these two methods have some disadvantages. For the solvent extraction method, it is difficult to separate sulfur compounds from the organic solvents and reuse these solvents. And by calcinations, sulfur compounds and aromatics are burned out, which can lose the heat value of fuels.

Another possible approach to produce ultra-low fuels is biodesulfurization (BDS) which can selectively remove sulfur from condensed thiophenes such as benzothiophenes and dibenzothiophenes without degrading the carbon skeleton of these organic sulfur compounds. BDS has the potential benefits of lower capital and operation costs and produces high valuable byproducts (5, 19).

We proposed a novel method to produce ultra-low sulfur diesel which is the integration of adsorption and biodesulfurization, that is, to regenerate desulfurization adsorbents with a microbial method (20). The effects of adding oil phase, cells concentration, volume of oil and ratio water-to-adsorbent were studied (20). Sulfur compounds can be adsorbed through different mechanisms such as direct M-S adsorption, π complexation, and van der Waals interactions. The interaction of sulfur compounds and

adsorbents is different. In this paper, desulfurization performances of different kinds of adsorbents such as AgY, reduced NiY, activated carbon and alumina, and bio-regeneration with selected *P. delafieldii* R-8 strains were studied. There are large amounts of aromatics in the diesel and these aromatics have a significant influence on the desulfurization performance and on the regeneration of adsorbents. Therefore, the effects of aromatics on the bioregeneration of desulfurization adsorbents were also studied.

MATERIALS AND METHODS

Chemicals

13X, NaY zeolite, and activated alumina were obtained from the Catalyst Plant of Qilu Petrochemical Company CNPC. DBT and naphthalene were purchased from Acros Organics, USA. 2-HBP was purchased from Tokyo Chemical Industry, Ltd. (TCI), Japan. *n*-Octane was purchased from Shanghai Reagents Co., China National Pharmaceutical Group Corporation. Methanol was HPLC grade; sodium chloride and ethanol was analytical reagent grade.

Adsorbents Preparation

Adsorbent AgY was prepared by the ion-exchange method according to the literature reported by Yang (21). AgY zeolite was prepared by ion-exchanging NaY with an excess amount of silver nitrate ($AgNO_3$) in an aqueous solution ($0.2\ mol \cdot L^{-1}$) at room temperature for 24–48 h in the absence of any source of light. The amount of silver content in the solution was equivalent to a 4-fold cation-exchange capacity. After ion exchange, the solid was recovered by filtration, washed with large amounts of deionized water, and then dried at room temperature also in a dark area.

Reduced NiY was prepared by ion-exchange method according to the literature reported by Song (22–24). Before using, NiY was pre-reduced in a flowing reactor under a reducing gas (5% H_2 and 95% N_2) with a flow rate $80\ mL \cdot min^{-1}$ at 640°C for 6 h, and then passivated using sulfur-free *n*-octane and stored in the same solvent in an airtight sample bottle.

Characterization

The temperature programmed reduction (TPR) measurement was carried out in a system supplied by Tianjin Xianquan Apparatus Co., Ltd., China (TP 5000). The samples were first palletized, crushed, and sieved to 150–250 μm size. During the TPR, the consumption of hydrogen was detected

using a thermal conduction detector (TCD). About 200 mg of the sample was loaded into a quartz reactor and heated at a rate of $10^{\circ}C \cdot min^{-1}$ in a stream of 5% H_2 in N_2 with a flow rate of about $80\ mL \cdot min^{-1}$ up to 900°C.

Desorption properties of the adsorbents were studied with thermogravimetric differential thermal analysis (TG-DTA) which was carried out in SP-4320 (Shanghai Precision Scientific Instruments Co., Ltd.) thermal balance. The sample was heated to 150°C in flow of inert gas (N_2) for 2 h to remove water and solvent *n*-octane. The spectra were recorded after maintaining the temperature for 30 min. About 20 mg of the adsorbents were loaded and the N_2 flow used was $50\ mL \cdot min^{-1}$. The heating rate was $10^{\circ}C \cdot min^{-1}$ and the final temperature was 700°C.

Bacterial Strain and Cultivation

P. delsfieldii R-8 (CGMCC 0570) was isolated from the sewage pool of the Shengli Oil Field of China (5).

P. delsfieldii R-8 was cultured in a standard medium (BSM) which has the following composition: KH_2PO_4 $2.44\ g \cdot L^{-1}$, $Na_2HPO_4 \cdot 12H_2O$ $12.03\ g \cdot L^{-1}$, $MgCl_2 \cdot 6H_2O$ $0.4\ g \cdot L^{-1}$, NH_4Cl $2.0\ g \cdot L^{-1}$, $CaCl_2$ $0.75\ mg \cdot L^{-1}$, $FeCl_3 \cdot 6H_2O$ $1\ mg \cdot L^{-1}$, $MnCl_2 \cdot 4H_2O$ $4\ mg \cdot L^{-1}$, glycerol $10\ g \cdot L^{-1}$. $1\ mmol \cdot L^{-1}$ DBT was added as the sulfur source. Cell cultivation was carried out at 30°C on a rotary shaker operated at 170 rpm.

Adsorption Methods

The adsorption capacity and selectivity towards DBT of the adsorbents were tested under ambient conditions with $8.0\ mmol \cdot L^{-1}$ DBT and $8.0\ mmol \cdot L^{-1}$ naphthalene as model compounds and *n*-octane as a solvent. The adsorption was operated at 30°C by mixing adsorbents with oil. The ratio of oil to adsorbent was chosen as $100\ mL \cdot g^{-1}$.

Regeneration of Adsorbent

Cells were harvested in the late logarithmic phase by centrifugation at 5000 rpm for 5 minutes. The cell pellets were washed twice with saline (NaCl, 0.75 wt%), lyophilized, and kept below −20°C.

The regeneration system contained *n*-octane, aqueous phase, lyophilized cells, and spent adsorbents. The ratio of water to adsorbent was chosen as $60\ mL \cdot g^{-1}$ and the Volume ratio of water to oil was 1/6 (V/V). Cells concentration was $25\ g \cdot L^{-1}$. All reactions were carried out in 100 mL flasks at 30°C on a rotary shaker operated at 200 rpm.

Recycling of AgY was studied with 15.0 mmol · L^{-1} DBT as a model compound. Ratio of oil to adsorbent was 40 mL · g^{-1}. The adsorption capacity of AgY was tested by mixing adsorbent with oil for 30 min.

Analytical Methods

High-performance liquid chromatography (HPLC) was used for the quantitative assay of DBT, naphthalene, and 2-HBP in the *n*-octane phase. HPLC was performed on a Agilent 1100 (HP1100, Agilent, USA) liquid chromatography equipped with an autosampler, a reversed-phase Zorbax SB-C18 column (4.6 mm × 150 mm; 3.6 μm) and a diode array detector. The mobile phase was 90% of methanol in water (v/v, %) with a flow rate of 1.0 mL · min^{-1}. For the quantification of the compounds, such as DBT, naphthalene, and 2-HBP the external standard method was used at 280 nm.

RESULTS AND DISCUSSIONS

TPR Study of Adsorbent NiY

The reduction of NiY was studied by TPR with reducing gas (5% H_2 and 95% N_2). As shown in Fig. 1, the reduction took place in two steps: with peak temperatures centering around 590°C and 730°C and the most intense peak appearing around 600°C. The first peak corresponded to the reduction of

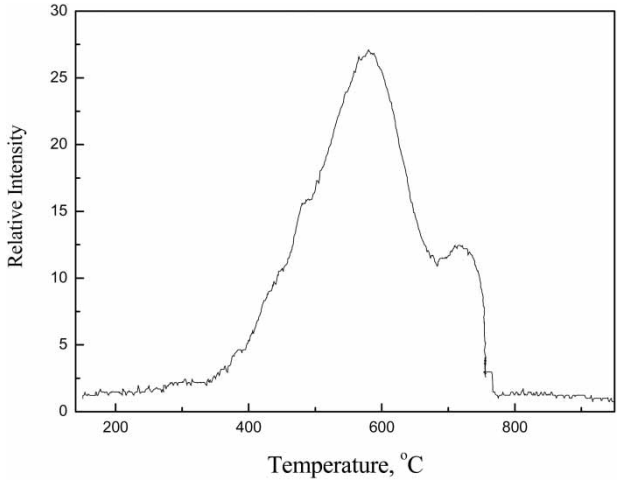

Figure 1. TPR spectra of NiY with reducing gas (5% H_2 and 95% N_2) at a heating rate of 10°C · min^{-1}.

Ni^{2+} to Ni^{+} and the second one to the reduction of Ni^{+} to Ni^{0}. Most of the Ni^{2+} ion-exchanged can be reduced around 600°C. Thus, with the given reducing gas, the optimum temperature for reduction of NiY was approximately 750°C. The large difference in intensities of the two peaks was likely caused by incomplete conversion of Ni^{+} to Ni^{0} because the reduction process is affected by the location and the amount of Ni ions exchanged in the Y zeolite. To ensure the complete reduction of Ni^{2+} to Ni^{0}, NiY should be left in the reducing gas for 3 h. The adsorbent NiY was prepared by reducing NiY at 750°C for 3 h.

Adsorption Process

Sulfur compounds can be adsorbed on the surface of adsorbents through π-complexation, van de Waals, and electrostatic interactions or reactive adsorption. Adsorption capacities such as adsorption capacity and selectivity of adsorbents 13X, alumina, AgY and reduced NiY were studied with 8.0 $mmol \cdot L^{-1}$ DBT and 8.0 $mmol \cdot L^{-1}$ naphthalene as model compounds. The ratio of oil to adsorbents was chosen as 80 $mL \cdot g^{-1}$. As shown in Table 1, the adsorption capacity of these adsorbents follows the sequence: activated carbon > reduced NiY > AgY > alumina > 13X. The selectivity of DBT over naphthalene follows the sequence: reduced NiY > AgY > 13X $\approx$ alumina > activated carbon.

TG-DTA Characterization

Desorption properties of DBT on different adsorbents were characterized by TG-DTA. Fig. 2 shows TG desorption profiles of DBT on different adsorbents. It can be seen that the adsorption capacity of DBT follows the sequence: activated carbon > NiY > AgY > Al_2O_3 > 13X. The interaction forces of DBT and adsorbents can also be concluded from Fig. 2. The activated

Table 1. Results of adsorption of DBT and naphthalene on different adsorbents

Adsorbents	Activated carbon	Reduced NiY	AgY	13X	Alumina
A_{DBT}, $mmol \cdot g^{-1}$	0.324	0.286	0.237	0.033	0.042
A_{Naph}, $mmol \cdot g^{-1}$	0.272	0.149	0.161	0.026	0.034
Selectivity	1.191	1.915	1.472	1.269	1.235

A_{DBT}: amount of DBT adsorbed per gram adsorbents;
A_{Naph}: amount of naphthalene adsorbed per gram adsorbents;
Selectivity: A_{DBT}/A_{Naph}.

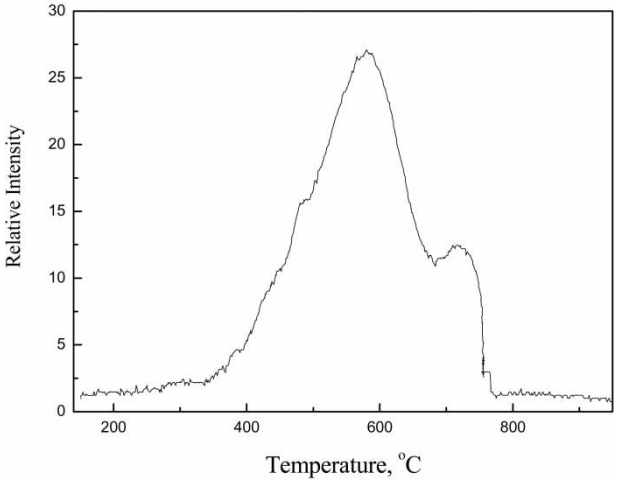

Figure 2. TG desorption profiles of DBT on different adsorbents.

carbon profile continues to decrease even when the temperature is higher than 400°C, but for that of 13X almost remains stable after 200°C. It can be concluded that the interaction of DBT with activated carbon is much stronger than that of DBT with 13X. The interaction of DBT with different adsorbents follows the sequence: activated carbon > NiY > AgY > alumina > 13X.

Bio-Regeneration Process

Desulfurization adsorbents can be the regenerated by the solvent extraction method and the bio-regeneration method. One of the advantages of bio-regeneration is that less extraction solvent was needed compared with the solvent extraction method. The effect of adding *P. delsfieldii* R-8 cells on the DBT desorption was studied with $60\,\mathrm{mL}\cdot\mathrm{g}^{-1}$ of water to adsorbent ratio and 1/6 (V/V) of water to oil phase ratio. From Fig. 3, the desorption ratio is higher when adding desulfurization cells. During the regeneration process, most of the DBT molecules adsorbed can be desorbed from the surface of the adsorbent by contacting with oil phase. Oil, water, and biocatalysts can be fully mixed by agitation and emulsified into micro-droplets at the oil/water interface. A part of DBT molecules can enter the aqueous phase directly. On the one hand, DBT molecules can be converted into 2-HBP and sulfate by microbial cells at the oil/water interface and in the aqueous phase. On the other hand, 2-HBP produced can enter the aqueous phase which can reduce the DBT concentration in the oil phase and promote DBT desorption

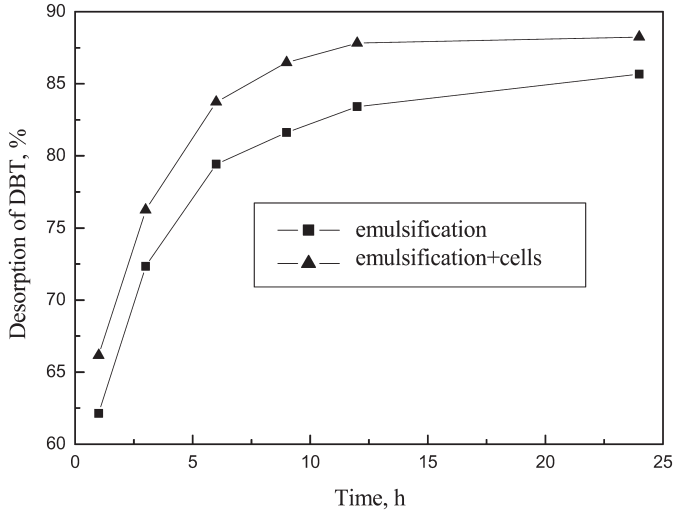

Figure 3. Effects of adding *P. delsfieldii* R-8 cells on the desorption of DBT.

from adsorbent AgY. Thus adding *P. delsfieldii* R-8 cells can improve DBT desorption from adsorbents AgY.

As we reported previously, during the process of bio-regeneration addition of the oil phase can significantly improve the desorption of DBT (20). In this paper, desulfurization adsorbents with different interaction with DBT were bio-regenerated when the volume ratio of oil-to-water was 1/5, the cell concentration was $50\ g \cdot L^{-1}$, and the ratio of water-to-adsobent was $60\ g \cdot mL^{-1}$. Figure 4 shows the desorption of DBT on different adsorbents by the microbial method. It can be seen that the desorption ratio of DBT follows the sequence: 13X > Alumina > AgY > NiYμ Activated Carbon. Comparing with the result of TG-DTA, it can be concluded that the desorption of DBT has a close relationship with the interaction of DBT with adsorbents. For activated carbon, the interaction of DBT with the adsorbent is so strong that only little DBT can be desorbed from the surface of the adsorbent.

Effects of Aromatics on Bio-Regeneration of Adsorbents

Aromatics have similar properties with organic sulfur compounds and can adsorb on the surface of adsorbents competitively with sulfur compounds. Aromatics in the fuels have important effects on adsorption desulfurization. During the process of bioregeneration, aromatics also have important effects on the desorption of DBT and on the regeneration of adsorbents. The effects of aromatics on the desorption of DBT were studied with AgY as

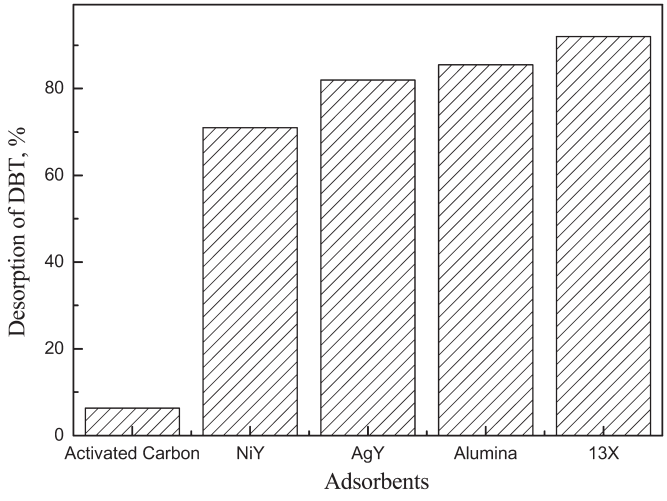

Figure 4. Desorption of DBT on Different adsorbents with microbial method.

adsorbent and DBT and naphthalene as model compounds. Adsorbent AgY was saturated by DBT *n*-octane solution and solution of DBT and naphthalene mixture (molar ratio 1:1) respectively. During the process of bio-regeneration, the volume ratio of *n*-octane and water is 1:10 and the same molar ratio of DBT adsorbed on the surface of adsorbents to oil phase was chosen. The effect of naphthalene on the desorption of DBT is shown in Fig. 5. The results show that when the DBT and the naphthalene adsorbed on the

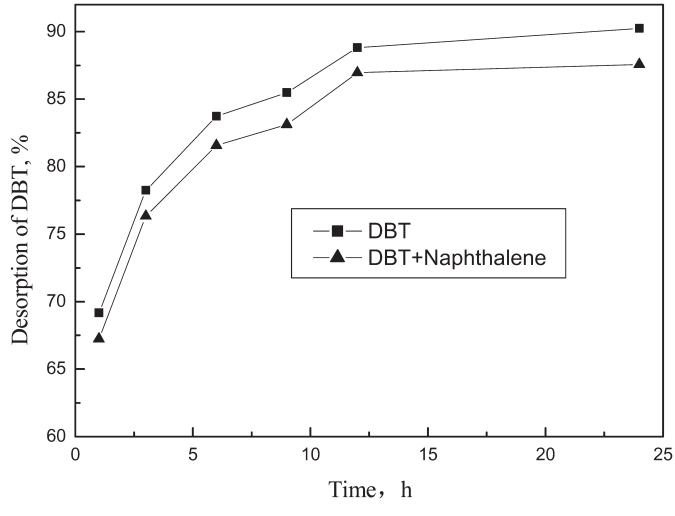

Figure 5. Effects of naphthalene on the desorption of DBT.

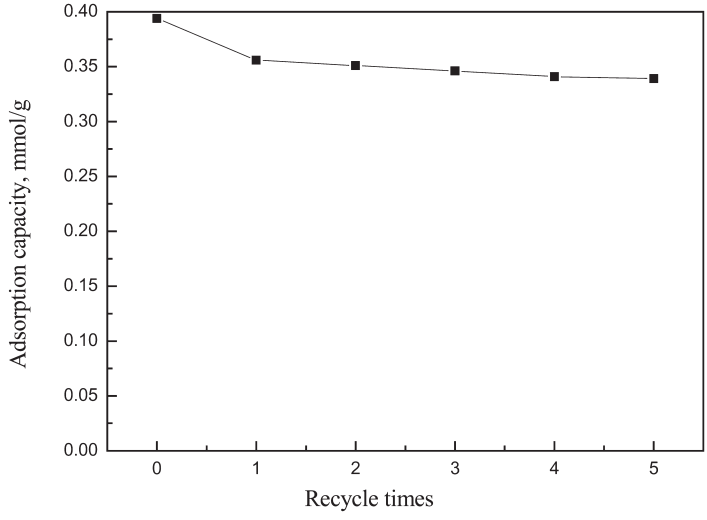

Figure 6. Effects of recycling times on the adsorption properties of adsorbents AgY.

adsorbents AgY simultaneously, the desorption of DBT decreased when compared with that where only DBT was adsorbed.

Recycling of AgY

Recycling of AgY was studied with 15.0 $mmol \cdot L^{-1}$ DBT as a model compound. Ratio of oil to adsorbent was 40 $mL \cdot g^{-1}$. As shown in Fig. 6, the adsorption capacity of AgY decreases for the first time recycling and then remains constant. Ion-exchange process can be reversed, that is, Ag^{+} can be replaced by Na^{+} present in the saline, which can cause the loss of Ag^{+}. Therefore, the decrease of adsorption capacity is due to the loss of Ag^{+} ions.

CONCLUSION

Adsorption properties of adsorbents reduced NiY, AgY, activated alumina, 13X, and activated carbon were studied with dibenzothiophene (DBT) and naphthalene as model compounds. Adsorbent NiY has the highest selectivity and activated carbon, the largest adsorption capacity. Adding *P. delsfieldii* R-8 cells can improve DBT desorption from AgY because DBT desorbed can be converted into 2-HBP with desulfurization cells thus improving the desorption of DBT. The desorption of DBT from adsorbents by bio-regeneration of adsorbents follows the sequence: 13X $>$ alumina $>$ AgY $>$ reduced NiYμ

activated carbon. Little decrease of the desorption of sulfur compounds can be caused by the presence of aromatics.

Ultra-deep desulfurization can be achieved by integrating of adsorption and biodesulfurization. During the process of bioregeneration, sulfur compounds can be desorbed efficiently from the many kinds of adsorbents except activated carbon and can be desulfurized without losing heat value with *P. delsfieldii* R-8. Desulfurization adsorbents can be reused. The adsorption capacity of AgY decreases for the first time recycling and then changes little. The decrease of the adsorption capacity is due to the loss of Ag^+ ions. The encouraging results of this work and the parameters investigated clearly point to a need for more detailed study with respect to various factors, such as the effect of the amount of aromatics, the separation of adsorbents and cells, and the particle size of adsorbents.

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