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Preparation and dye adsorbing properties of Fe 3O4 carbon/graphene oxide composites.

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Abstract: Fe3O4@carbon/graphene oxide (Fe3O4@c/go) composites were synthesized by hydrothermal method and characterized by X-ray diffraction, transmission e Lectron microscopy, vibrating sample magnetometer. And their dye

Adsorbing properties were investigated. The results indicated that the Fe3O4 Nanoparticles were uniformly anchored on go sheets. The saturated adsorption capacities towards rhodamine B (RhB) of the composites increased with the increase of Go content, initial RhB concentration and Adsorbing temperature. And the higher go content in The composite was, the quicker the absorbing Rate was. The saturated adsorption capacity of The composite reached a maximum value When the PH value is 7. For the composite prepared at 0.8 Of the mass ratio of Go To Fe 3O4, The saturated adsorption capacity reached 303.4mg/g at 1000mg/l of initial RhB concentration.

Keywords: Fe3O4; Carbon ; Graphene oxide ; Dye ; Adsorption

Dye Pollution band Water Body Ann Full Issue Primer Wide Focus[1].Dye Material Waste Water Place rationale Many methods , such as biological method, away Sub -interchange, photodegradation , chemicalflocculation and adsorption[2-4]. where , suction with the method commonly used in the the the hard to live things drop solution Dirty dye , and and also waste water Depth The Important technical operation . Suck The excellent decision of the attachment Adsorption Processing Effect Fruit . Research Investigate report Suction agent has carbon nanotubes[5], Clay Minerals[6]Fly ash[7], polymeric resins[8], Biochar[9], and so on . How to get Excellent Good adsorption Agent still related Domain Research Focus and Hot Point . Magnetic Suction attached material material due to both good Suction Attach Force , and With Dirty Dye Fast Detach

attributes , caused people's wide generics note [10-12].researchers prepare by various means F e3O4@Carbon (Fe 3O4@c) Duplicate Composite Material , and carried out the water in the smoke suction The attachment study . Research investigate Result Table Ming , Fe3O4@C duplicate material suction attach performance with its surface area, hole diameter, and F e3O4 The structure is related to the dosage[12-13]. F e3O4@C composite materials are readily accessible , non-toxic , and synthetic methods simple

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single Feature , and the adsorption process does not introduce harmful to water bodies Substance , become an

important adsorbent material.

Graphene as to a new Carbon material because of its giant larger than table area and Good Good surface can Cosmetic Make its can be an excellent Good Suck Attach Material Material[14-16],but face Pro Difficult to back Collect, Heavy Compounding Low usage issues , and the F e3O4 after composite , then give it good magnetic sex off and back accept again Health special sex , can toGive full play to two by Excellent Potential . Research people mining with solvent hot Method and water Hot method etc side preparation of graphene oxide /Fe 3O4 Composite Material (go/fe 3O4), andDye Adsorption Research investigate , its Result Table Ming blending material to dye the material has a good adsorption capacity[17-19].

This article is Go , coprecipitation Fe3O4 and soluble starch as raw material , prepared by hydrothermal method Fe3O4@c/go Composite Materials . starch basal material carbon will Fe3O4 Wrap and fix to Go Flat . this keeps the Go larger than biomass carbon tables

Nitrogen Deaerator 15min;2) The resulting mixture transferred to the hydrothermal response Kettle , Heat to 180 ℃ response 20h;3) after reaction ends , products washed and ethanol washed several times , Vacuum dry to be used.

1.3 Fe3O4@c/go Material adsorption Rhodamine B Experiment

0.05g adsorbents dispersed in 100mL different concentrations of RhB solution , timed sampling , remove supernatant after separation of magnetic fields , mining with ultraviolet visible spectrophotometer (TU1810, general analysis of Beijing instrument have limited responsibility no Company) on RhB most suck Accept wave long 552nm measure its concentration . The amount of adsorption at different times q T(mg/g) calculation by the next:

QT=100 (C0-CT)/m (1)

In-style :C 0 For dye initiation concentration , mg/l; C T The remaining concentration after adsorption , mg/l M for adsorbent quality , G. The same calculation of saturated adsorption quantity[15].The experiment flowchart is shown in the figure 1 .

Coprecipitation method

Area and porous structure characteristics , and can stabilize Fe3O4,Make it not

Easy to fall off , and not be oxidized to lose magnetism , thereby expanding
application range of composite materials . The structure and RhB adsorption performance studied.

1. materials and methods

Uv-vis Detect

Magnetic separation

1.1 Experimental material

ferric chloride AR grade, sodium hydroxide ar level, soluble lakes pink ar level , Beijing Modern Oriental Fine Chemicals Co., Ltd. ; Ferrous sulfate AR level , rhodamine B (AR level), Tianjin Guangfu Fine Chemical Research Institute ; Go (self -made)[20].

1.2 Fe3O4@c/go Preparation of Materials

F e3O4 nanoparticles Pass Total Precipitate Method Prepare : 1) Call FeC L3·6 H2O (40mmol) and F e S O4·7 H2Ommol) dissolve in 300mL go to Ionic water , NaOH (+) 0m m ol) also dissolve in 300mL deionized water (Fe 3+: Fe2+: OH- =2:1: 8),

With a solution through the nitrogen 30min to remove the Oxygen 2) with constant current pump will Fe ion solution to join NaOH Solution , flow rate 5ml/min, react immediately after blending , The entire process continues to be nitrogen-guaranteed protect ; 3 after , the products obtained with water and ethanol wash several times , until PH to Neutral , then 60 ℃ Vacuum drying 24h, get Fe3O4 Nanoparticles.

prepared by hydrothermal method Fe3O4@c/go Material : 1) Call

prepared 0.65g Fe3O4, 3g soluble starch and a certain amount of

Go , Join 50mL deionized Water , Ultrasonic processing 10min, and Charge

1.3 Material Characterization

using Japanese philosophy X X-ray Diffractometer (Riguka d/max-2400 type , excitation source is cuka target ,λ =0.15418nm, 40kv,200ma, Graphite Monochromator , X - Ray Count is blinking Digital Device) material phase analysis ; using transmission electron microscopy Mirror (TEM, hitachi H-? type) characterize the microstructure of the adsorbent and morphology ; using Vibrating sample magnetometer (United States Lakeshore 7307vsM) Test Sample , room temperature Magnetic hysteresis Line ; Take bk100c type specific surface area and porosity Analyzer (Beijing subtle Gaobo Science and Technology Co., Ltd.) make N2 adsorption - desorption experiment , through BET (Brunauer-Emmet-Teller) model Calculation sample product than surface product , through DFT (Density functional theory) theory to calculate aperture distribution ; Take escalab , type X X-ray photoelectron spectrometer (United States THERMO VG company) Analysis of the content of material elements and surface oxygen groups;

with zetapals type Zeta potentiometer (American Brookings Instrument Co., Ltd) test and determine the electrical point of the material.

2. esults and discussions

2.1 adsorbent Structure

Figure 2 to Sample X RD diffraction curve Line . Fe3O4 Of diffraction diagram in . 0 (35.2), 311, 42.9 (), 56.9 (511)

and 62.5 (440) has diffraction peak , and anti-sharp crystal stone structure

Fe3O4 - (Jcpds card number -086 3) Consistent Data , not

Miscellaneous Peaks , Peak and Sharp . Go has a wide on 24.8

diffraction Peaks . Fe3O4@c the diffraction curves of composite materials can only be seen

Obvious Fe3O4 diffraction Peaks , description of the carbon components included is amorphous

structure , Fe3O4@c/go Composite in 24.8 appears around

Go diffraction Peaks , And the intensity of the diffraction peaks with Go Add Quantity

Increase and increase.

Figure 3 for Sample TEM Photo . Coprecipitation Synthesis of Fe3O4 the mean particle size of the nanoparticles is 12nm, after carbon wrap Fe3O4 particle dispersion evenly . **Figure 3** (b) - (d)to different go/Fe 3O4 prepared with matching conditions Fe3O4@c/go Composite TEM Photo . Show Results , Fe3O4 nano-particles loaded in a sheet-shaped structure Go Top , and with Go Increase in usage , Monolithic Go on Fe3O4 nano-particles reduction . samples undergo lengthy ultrasound processing during cleaning , but Fe3O4 particles still well loaded Go Without shedding . The result clearly indicates , the coating of starch based carbon and its associated with Go A good combination of Fe3O4 particles firmly anchored in Go Top.

Figure 4 the is the hysteresis curve at the sample room temperature . prepared material all with super smooth Magnetic properties , Fe3O4 Nano particle , Fe3O4@c and Fe3O4@c/go (go vs . Fe3O4 quality ratio The magnetic saturation intensity of the to 0.8) is as follows, Ms , respectively 56.1,15.1 and 11.9emu/g. Fe3O4 with biomass carbon and go after composite ,its relative content down , The magnetic saturation strength of the composite is reduced by low . if Fe3O4 nanoparticle content is too low , Composite Ms will be too small ,thereby weakening the magnetic separation capability of . **Figure 4** The illustration in clearly shows the , prepared Fe3O4@c/go (same on) can be quickly and easily separated from water by the external magnetic field out.

Fe3O4@c/go (go and Fe3O4 quality ratio to 0.8) nitrogen Adsorption - desorption curve as pictured 5 shown , has a noticeable hysteresis loop . This indicates that the material has a medium hole structure . aperture map Results Display , the material is mainly medium hole and large pore structure . the specific surface area of the material and

total porosity, respectively 62.5m2/g and 0.343cm3/g, Micro Conconjon to 0.024cm3/g. also , Fe3O4@c specific surface area and total hole tolerance is 15.6m2/g and 0.069cm3/g, Micro Conconjon to0.0045cm3/g. By comparison , Go The addition of the increases the specific surface area and porosity of the composites , significantly improved performance.

F e3O4@c/GO Duplicate Fit Material Material Table face Power Sex is by its surface H+, O H-charged Electric ion-determined . when PH value is small when Power Point , The material surface is positively charged ; when PH when the value is greater than the power point , The surface of the material is negatively charged.

XPS diagram 0.8 . The results indicate that , composite surface ,Fe content is 0.54wt.%,o content is 24.66wt.%,c content is 74.8wt.%, result analysis shows , composite surface has rich oxygen-containing functional groups , as table As shown in 1.

Figure 7 Medium Fe3O4@c/go Composite Zeta Potential Map . Fe3O4@c/go the electrical point of the composite is about ph=2.

Figure 7 Sample Zeta potential and PH Relationship of Values

fig.7 relationship between Zeta potential and PH value of sample

2.2 Go and Fe3O4 The effect of the ratio on the adsorption performance diagram 8 for material RhB Isothermal Adsorption curve . Clear Results

Show , Composite RhB saturated adsorption volume with Go with

Increase in volume , when W (GO)/ W (Fe3O4)more than 0.8 after , There is little change in saturated adsorption , But the adsorption rate increases . Pure Fe3O4 Basic no adsorption capability , Fe3O4@c composite saturated adsorption only 15mg/g, but W (GO)/ W (Fe3O4)to 0.8 and 1.2 prepared under conditions Fe3O4@c/go the saturated adsorption values of composites are 48.5mg/gand 48.7mg/g. The results clearly indicate , Go the introduction of the significantly enhances the material pair RhB Adsorption capacity . from cost and magnetic recovery efficiency perspective ,Select appropriate W (GO)/ W (Fe3O4)ratio can . follow up experiments with W (GO)/ W (Fe3O4)to 0.8 products prepared under conditions.

2.3 effect of initial dye concentration on material adsorption properties

Figure 9 The shows the effect of initial dye concentration on material adsorption properties . with RhB increase in initial concentration , Fe3O4@c/go increased saturated adsorption . RhBInitial concentration from 25mg/l up to 1000mg/l when , Fe3O4@c/go the saturated adsorption value from 33.7mg/g increase to 303mg/g. Composite initial adsorption rate is fast , in 30min The basically reaches saturation adsorption . Initial adsorption , Fe 3O4@c/go more

adsorption sites on material surfaces , so suck attach speed , decrease with adsorption sites , inside and outside the material RhB Decrease in concentration difference , The absorption resistance increases gradually , RhB contact with the adsorbent opportunity to decrease , so the adsorption rate is lower[21].Composite materials can The reason for adsorption RhB may be : 1 RhB for cationic alkaline dyes , can be used to create static effects with materials ; 2) RhB has carboxyl groups , can be with Fe3O4@c/go hydroxyl and carboxyl groups on the surface produce hydrogen bonds ; 3) conjugate of both π key structure causes each other π-π Stacking effect[22].

Figure Ten Temperature pair Fe3O4@c/go Adsorption RhB Effects fig.10 Effect of temperature on RhB adsorption of Fe3O4@c/go

temperature is an important factor affecting adsorption . Figure Ten saturated adsorption for composites at different temperatures . Fe3O4@c/go to dye material adsorption physical adsorption and chemical adsorption . Physical Adsorption process faster , Strong adsorption , for reversible adsorption , temperature rise Easy resulted in the removal of . The chemical adsorption is made by the adsorbent and the adsorbed mass. Learning key forces for , adsorption heat greater , adsorption requires activation can , temperature increases for adsorption[23].also , temperature is sticky to solution degree and dye molecular activity also affect , within a certain range , with temperature rise , solution viscosity Decrease ,Molecular motion intensifies , and The adsorbent has an increased chance of collision , which in turn facilitates adsorption , make adsorption

Increase . Experimental results show , in 30~70 ℃ , increase with adsorption temperature , increased saturation adsorption of adsorbent , in $ ℃ reached 97.9mg/g, adsorption should be mainly chemically adsorbed [24]. 2.5 PH value effects on material adsorption properties

Figure One to different PH value conditions for composites RhB Saturated adsorption . saturated adsorption with PH increase the value first and then lower , in PH value is 6.94 when maximum is reached (67.4mg/g). PH value in 2.01~6.94 in scope , Fe3O4@c/go surface containing oxygen groups to the degree of proton gradually increased , the makes the adsorbent and RhB stronger force of molecules ; and in 6.94~11 in scope , PH when values continue to grow , RhB reunion occurs , that is, the transition from a single molecule to a two-molecule binding body , Increased adsorption resistance , therefore saturated adsorption decreased by . The results above also indicate , if at a lower PH the adsorbent can be reclaimed for regeneration[25]. PH The effect of the value on the adsorbed process may be mainly the oxygen-containing functional group on the surface of the adsorbent and the structure of the dye molecule [26]. The XPS test concludes with , Fe3O4@c/goThere is a large number of oxygen-containing groups on the surface of materials , bring its surface Negative Charges . At the same time through the material in different PH under value conditions Zeta The electric potential test draws its equal point to the 2, when dye solution PH value is greater than 2 when , The surface of the adsorbent is negatively charged , facilitates the adsorption process . To summarize , adsorbents and RhB primary role between molecules

It's electrostatic absorption..

Layer , Composites can be separated quickly from water by an external magnetic field out.

Fe3O4@c/go Composites on dyes (RhB) has Good adsorption , with go Increase in dosage , the adsorption rate and saturated adsorption of the composites increased with the..

in experimental scope , Fe3O4@c/go to RhB the saturation adsorption of the with temperature and RhB increase in initial concentration , with PH Increase first and then lower , in PH value is 6.94 when maximum is reached.

Fe3O4@c/go rich in oxygen-containing functional groups on the surface , its power point PH value is 2, and RhB the role of the molecule is mainly electrostatic adsorption , and hydrogen bonding.

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