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泡沫陶瓷基氢氧化高镍催化剂的制备及模拟污水处理研究

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摘　要: 本研究以活性炭为造孔剂制备泡沫陶瓷,通过浸渍法使 $NiCl_2$ 负载于泡沫陶瓷,再与 NaClO 和 NaOH 混合溶液反应生成 NiOOH 催化剂. 以一定浓度的亚甲基蓝为污水模拟物,亚甲基蓝褪色时间来衡量 NiOOH 催化剂的催化性能. 以催化剂量、次氯酸钠量、pH、温度为变量,分别通过单因素实验和正交试验探究了它们对催化效果的影响以及确定污水处理的较优组合条件. 结果表明,在温度为 55 ℃、pH 为 8、催化剂量为 1.0 g、NaClO 量为 15.0 mL 时,亚甲基蓝的完全褪色时间为 131 min. 各因素对污水处理效果的影响顺序为温度>催化剂量>pH>NaClO 量. 污水处理的最佳实用组合条件为温度为 35 ℃、pH 为 8、催化剂量为 1.0 g、NaClO 量为 15.0 mL.

关键词: 泡沫陶瓷; 镍催化剂; 次氯酸钠; 新生态氧[O]
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Study on preparation of ceramic foams-based nickelic hydroxide catalyst and simulated sewage treatment

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Abstract: Activated carbon was used as the pore forming agent to prepare ceramic foams on which $NiCl_2$ was loaded by impregnation, followed by reaction with NaClO and NaOH mixed solution to generate NiOOH catalyst. Methylene blue was employed as the sewage simulacrum, and the catalytic performance of NiOOH catalyst was measured by fading time of methylene blue. The dosages of catalyst and sodium hypochlorite, pH, and temperature were used as variables to explore the influences on catalytic effect of sewage treatment and determine the optimal combination conditions by the single factor and orthogonal test, respectively. The results indicate that the complete fading time of methylene blue is 131 min when the temperature, pH, dosages of catalyst and NaClO are 55 ℃, 8, 1.0 g and 15.0 mL, respectively. Sequence of significant effect of factors on fading time of methylene blue is temperature > dosage of catalyst > pH > dosage of NaClO. The optimal practical combination conditions for sewage treatment are

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confirmed as that the temperature, pH value, and the dosages of catalyst and NaClO are 35 ℃, 8, 1.0 g, and 15.0 mL, respectively.

Keywords: Ceramic foams; Nickel catalyst; Sodium hypochlorite; New eco oxygen [O]

1 Introduction

Water resources are the indispensable for human survival, and the rational utilization and recycling of them become an urgent problem to be solved. As the sewage treatment technology in our country started relatively late, it has become an important factor affecting the development of sewage treatment industry. It is reported that sodium hypochlorite is widely used in various sectors of the national economy as a bactericide, a bleach and an oxidant[1]. In social life, especially in the treatment of urban sewage, sodium hypochlorite has been widely used because of its good disinfection and simple operation[2] due to its low price and small pollution[3]. The principle is that a large number of new ecological oxygen [O], which is a powerful oxidant, was produced in the decomposition process of sodium hypochlorite. This performance plays a key role in the process of sterilization, bleaching, *etc*[4]. The speed and degree of sterilization and bleaching are positively correlated with the formation rate and the concentration of new ecological oxygen [O]. Sodium hypochlorite solution is unstable, and the decomposition is easily influenced by many factors such as light, temperature, concentration, pH, and impure metal cations[5]. However, the decomposition speed of sodium hypochlorite under natural conditions is slow, and thus the oxidation efficiency is quite low. How to improve the decomposition rate of sodium hypochlorite is particularly critical. It has been shown that catalytic method can increase the decomposition speed of NaClO and improve its oxidation performance. It can be considered as one of the most effective ways to treat organic wastewater[6,7].

At present, studies on impacts of the decomposition of sodium hypochlorite are relatively rarely reported. Decomposition of sodium hypochlorite is caused mainly by transition metal ions such as Cu, Ni, Fe, Zn and Pb, among which Ni and Cu are significant[8]. Nickel can be considered as a catalyst because of its good dispersion and easy loading. A redox reaction can be promoted by Nickel catalyst due to its variable valence[9]. In a series of previous work, Ni_2O_3 has been used to produce [O] by decomposition of sodium hypochlorite, and appreciable results have been achieved[10]. Besides, Nickel catalyst was also used to catalytic decomposition of Calcium hypochlorite[11]. However, in industrial production, it is difficult for recovery of the catalyst with a low concentration after the addition of catalyst to wastewater due to the difficulty in solid-liquid separation. As a result, it is necessary for the loading and dispersing of Nickel catalyst on a carrier. Nickel catalyst has a good selectivity and activity at a low temperature, but its activity is affected by the properties of carrier materials[5]. So, it is critical for choosing a suitable carrier to support the catalyst. The most studied carrier materials are activated carbon, alumina, molecular sieves and zeolites, ceramic, and silica gel, *etc* [6]. Besides, it has been reported that foamed cement was used to support Nickel catalyst to accelerate decomposition of sodium hypochlorite to treat practical printing and dyeing wastewater[7,12,13]. However, in the foamed cement, the pores are mainly closed, and the specific surface area is quite small, which is not conducive to effective loading of catalyst.

Ceramic foams, one of the most important materials, have been widely used in a range of fields, namely filters, catalysts supports, bone scaffolds, due to their characteristics of high permeability, high porosity and specific surface area[14-16]. Combining with our previous work[17-20], ceramic foams were prepared by adding activated carbon as the pore forming agent and used to sup-

port Nickel catalyst. The simulated sewage treatment, which was added sodium hypochlorite in advance, was carried out with methylene blue as the simulated pollutant. It is convenient for recycling, and can effectively prevent secondary pollution. This work may provide a new way for investigation of sewage treatment.

2 Experiment

2.1 Preparation of ceramic foams

Kaolin, talc powder, and alumina with a mass ratio of 24∶8∶3 were mixed uniformly to be ceramic raw materials, and activated carbon particles with the diameter of 0.3-0.6 mm were used as the pore forming agent. The ceramic raw materials and pore forming agent in mass ratio of 1∶1 were mixed evenly, adding a certain amount of polyvinyl alcohol solution (PVA 1788, 8.5 wt.%). The mixture was mixed uniformly, followed by molding in a mould sized (21±1) mm×(21±1) mm. Subsequently, they were placed to dry in air atmosphere at room temperature, and then put in the oven to further dry at 80 ℃ for 24 h. After that, they were demoulded to get ceramic foams green body, followed by calcination in a furnace at 1200 ℃ for 2 h in air atmosphere. The samples were cooled down to room temperature in the furnace to obtain the final ceramic foams.

2.2 Nickel catalyst loaded on ceramic foams

Ceramic foams were cut into small hexahedral pieces with a size about 5 mm×5 mm×5 mm and dipped in the $NiCl_2$ solution (0.5 mol/L) for 2d, 4d and 6d numbered 1, 2 and 3, respectively. After immersion, they were dried at 80 ℃ in the oven, and then cooled down. The aforementioned $NiCl_2$ particles loaded on ceramic foams were impregnated in the mixed solution of excess NaClO and NaOH for 24 h, followed by drying at 80 ℃ in the oven to get the black Nickelic hydroxide (NiOOH). The specific chemical reaction is shown in Formula 1.

$$2NiCl_2+NaClO+4NaOH\rightarrow 2NiOOH+5NaCl+H_2O \quad (1)$$

2.3 Simulated sewage treatment

Methylene blue solution with a concentration of 10 mg/L was prepared and used in all of the experiments to simulate the sewage (200 mL per group). After adjusting pH value, methylene blue and sodium hypochlorite were treated in a water bath at the desired temperature, respectively. The two solutions were keeping for 10 min seperately, and then mixed uniformly. After that, Nickel catalysts loaded on ceramic foams were added into the above mixed solution, and the complete fading time of methylene blue was recorded to be the evaluation index.

By referring to similar studies in literature[13], the temperature, pH, and dosages of catalyst and NaClO were set to be 35 ℃, 8.0, 1.0 g and 15.0 mL, respectively as the benchmark for the single factor experiment to study the effect of them on the fading time of methylene blue, as shown in Tab. 1. In addition, in order to determine the sequence of significant effect of factors on the decomposition of sodium hypochlorite and the optimal condition for decomposition of sodium hypochlorite, an orthogonal test of four factors at three different levels was designed in this study. The four factors are the temperature, pH value, the dosages of catalyst and sodium hypochlorite, and the corresponding three levels are shown in Tab. 2.

Tab. 1 Design of single factor experiment

Catalyst dosage (g)	Temperature (℃)	pH value	Sodium hypochlorite dosage (mL)
0.0	15	3.0	5.0
0.5	25	5.4	10.0
1.0	35	8.0	15.0
1.5	45	10	20.0
2.0	55	12	25.0

Tab. 2 Design of the orthogonal test

	Temperature (℃)	Catalyst dosage (g)	pH value	Sodium hypochlorite dosage (mL)
Level 1	25	0.5	5.4	10.0
Level 2	35	1.0	8.0	15.0
Level 3	45	1.5	10	20.0

2.4 Characterization

The open porosities (P) of ceramic foams are determined by the Archimedes method. The microscopic structures of the specimens before and after loading were characterized by a scanning electronic microscopy (SEM, VEGA3, TESCAN, Czech Republic). The crystalline phases of the samples were investigated by an X-ray diffractometer (DX-2700, Dandong Haoyuan Instrument Co., Ltd., China) using Cu Kα radiation at a scanning rate of 0.05° s^{-1} and a working voltage/current of 40 kV/40 mA.

3 Results and discussion

3.1 Open porosity analysis

The open porosities (P) of ceramic foams are determined by the Archimedes method[17,18]. The open porosity of foamed ceramic is listed in Tab. 3. It can be seen that the open porosities of the 5 parallel samples in the experimental group have little difference, and all of them are maintained between 42%～46%. In order to avoid the accidental, an average open porosity of 43.53±2.47% was used as the eventual value. The reason for the low obtained open porosity is that the pore forming agent content is not high (50 wt.%). The pore forming agent (activated carbon) occupies a certain volume, and is removed forming carbon dioxide by calcination and leaving the pores. The dosage of pore forming agent and particle size play a dominant role in deciding the porosity and pore size.

Tab. 3　Open porosities of ceramic foams

Samples	m_1(g)	m_2(g)	m_3(g)	Open Porosity (%)
1	7.3094	10.0995	6.5481	42.61
2	6.9984	9.8580	6.2163	46.00
3	6.9061	9.5052	6.0913	42.67
4	6.3535	8.7283	5.6355	42.14
5	6.9502	9.6428	6.0890	44.22
Average value	--	--	--	43.53 (±2.47)

3.2 SEM analysis

Scanning electron microscopy (SEM) was used to investigate the morphology of ceramic foams, and Nickelic hydroxide loaded on ceramic foams before and after simulation of wastewater treatment. Fig. 1 shows the SEM images of raw ceramic foams at different magnifications. It can see from Fig. 1, a rugged surface of ceramic foams is existed at low magnification. There are many pores distributed unevenly and interconnected throughout the three-dimensional channels. The pore walls are relatively smooth, and the macropores are formed due to the burning out of activated carbon. It can be observed that some independent small holes are distributed on the hole struts at high magnification. It is because, on one hand, the binder was burned out forming small pores in ceramic foams after calcination; on the other hand, some small activated carbon particles were burned out.

Fig. 2 is SEM images of $NiCl_2$ loaded on ceramic foams by dipping 2d, 4d, and 6d. Comparing with the original foamed ceramic in Fig. 1, the pores are still interconnected with each other through the three-dimensional network channels at low magnification. However, the wall and struts of pores are not as smooth as that of the original ceramic foams. Instead, the pore wall and struts are covered by some materials and become rough. At high magnification, a dense and irregular crystal structure on the pore wall can be observed.

Fig. 3 presents SEM images of NiOOH loaded on ceramic foams by the reaction of $NiCl_2$ (2d, 4d, and 6d) with NaClO and NaOH. The pore wall and struts are still rough at low magnification. The loading became smoother at high magnification as compared with that in Fig. 2, which is due to reaction (1).

After reaction, the green $NiCl_2$ crystals were transformed into the black NiOOH particles attached unevenly to the pore surface and struts. For the sample of 4d, obvious flake and irregular granular crystals are observed on ceramic foams, which may be a by-product of the transformation from the green $NiCl_2$ into the black NiOOH.

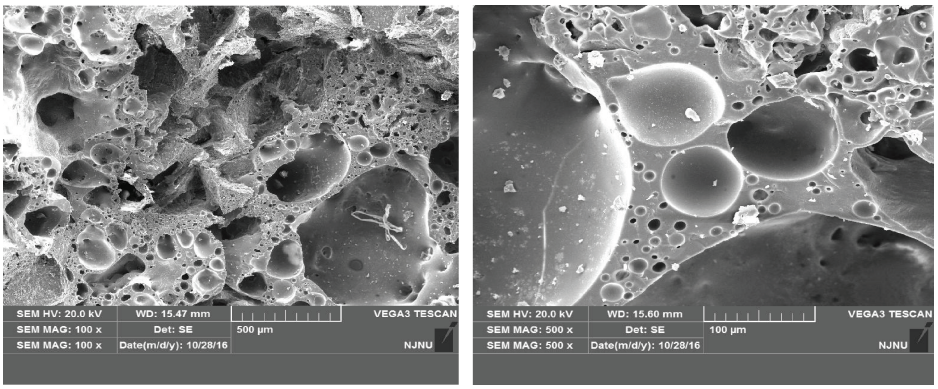

Fig. 1 SEM images of ceramic foams at different magnifications

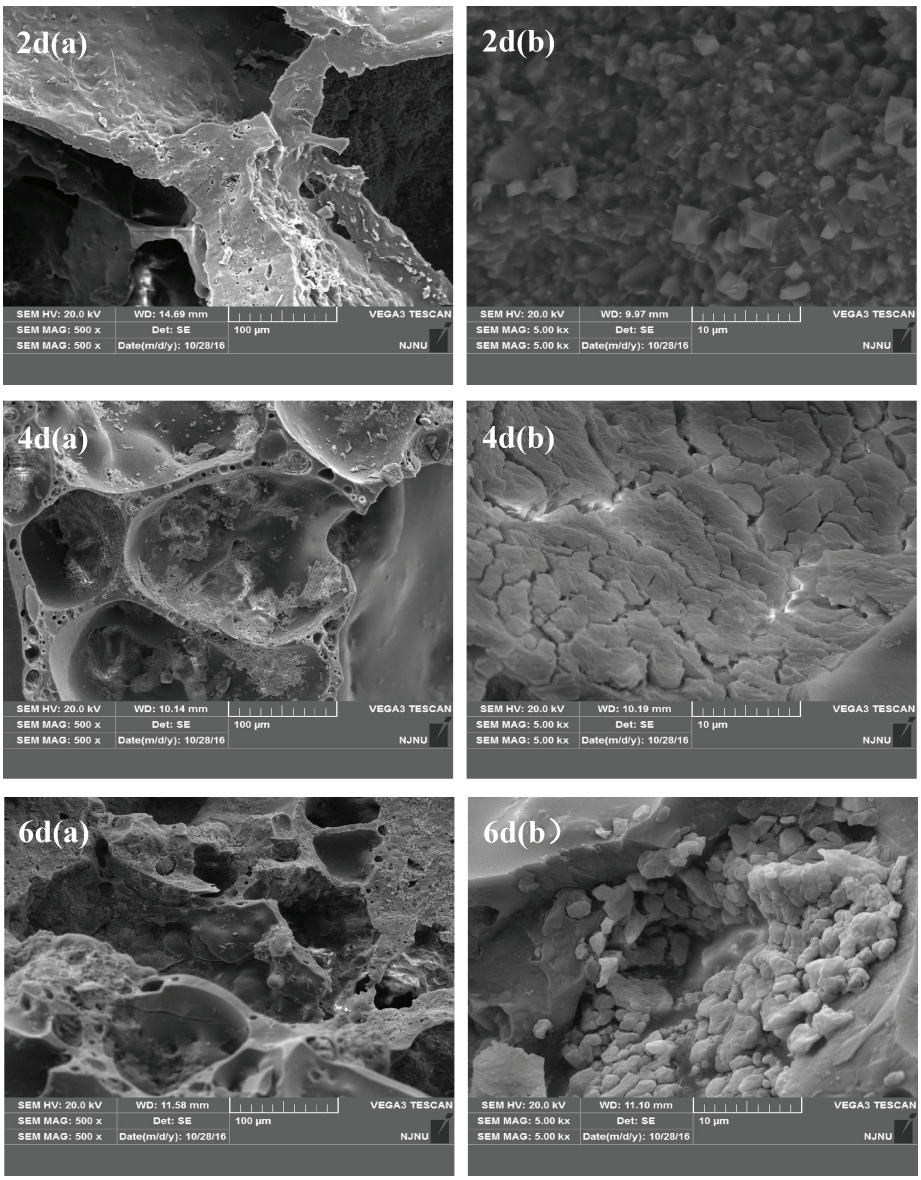

Fig. 2 SEM images of $NiCl_2$ loaded on ceramic foams

Fig. 4 is a SEM image of NiOOH catalyst loaded on ceramic foams after simulated sewage disposal. It shows that the irregular loads are still existed on the inner wall of ceramic foams after the sewage treatment. The microstructure of the samples of 2d and 6d at high magnification are in

between Figs. 2 and 3, and the inner wall of ceramic is rough. Some load can be seen roughly granular, and some load is unshaped. The microscopic image of 4d sample is obviously different from the other two samples. A mixture of angular cubic crystal and granular crystals are existed, which may be associated with the conditions of simulated sewage treatment. This group experiment was carried out at a pH value of 5.4, and the reagent used to adjust pH was hydrochloric acid. The crystallized powders of $NiCl_2$ and cubic crystal of NaCl were generated on the surface of ceramic foams.

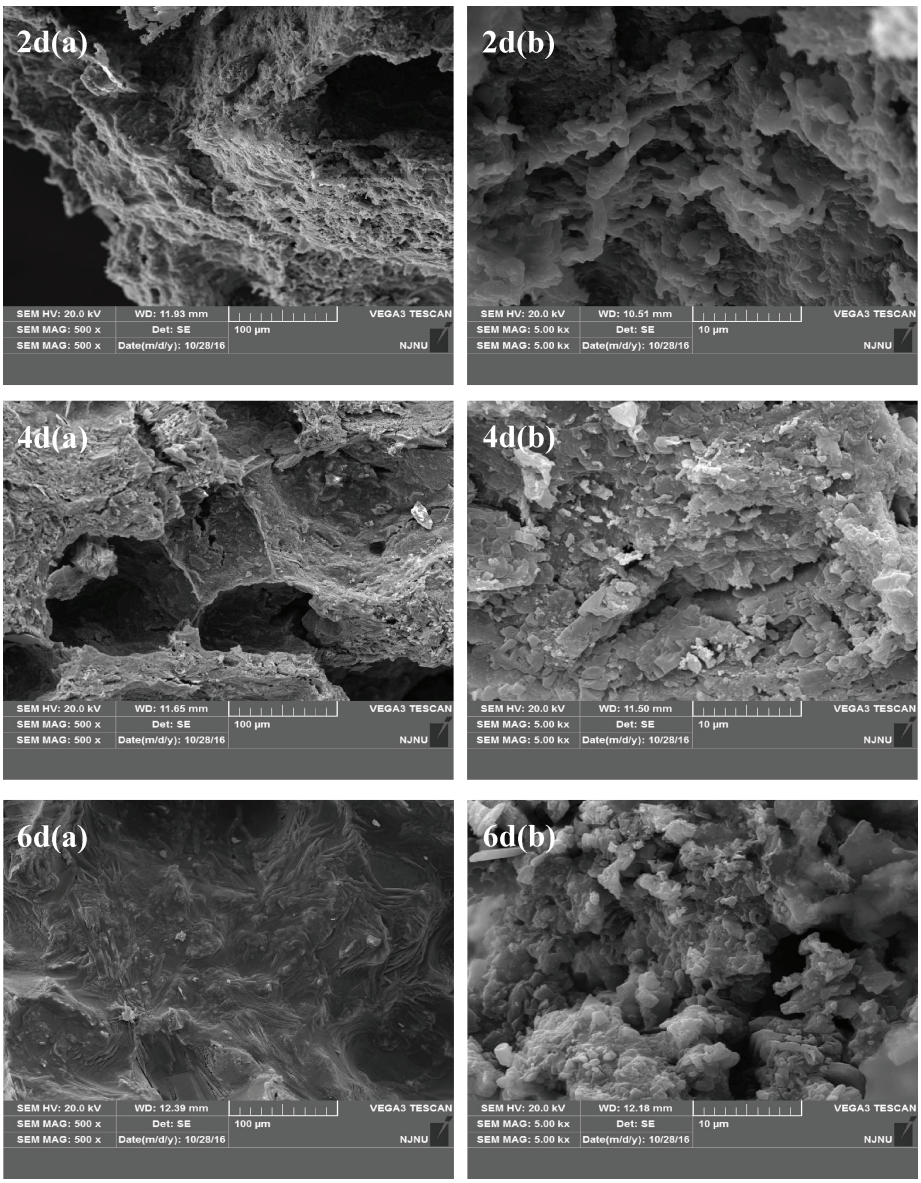

Fig. 3　SEM images of Nickelic hydroxide loaded on ceramic foams

It can be seen from Figs. 2－4 that, on the whole, impregnation days have little effect on the loading surface of ceramic foams, and the intensive loads appear on the pore surface. Considering the actual conditions, two days are chosen as the ideal immersion time of foamed ceramic to save time and improve the efficiency of sewage treatment. At the same time, a load was still existed on the pore surface of ceramic foams through the observation of the sample after simulation of sewage treatment. Therefore, Nickel catalyst loaded on ceramic foams can be considered reusable.

3.3 XRD analysis

Fig. 5a is the XRD pattern of ceramic foams. It can be seen that the diffraction peaks of mullite ($3Al_2O_3 \cdot 2SiO_2$), corundum (Al_2O_3), and quartz (SiO_2) are observed in ceramic foams which do not support catalysts. Cristobalite is from the raw material kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$), and corundum is from alumina (Al_2O_3) and kaolin. Kaolin was decomposed into free SiO_2 and Al_2O_3, and thus the diffraction peaks of kaolin disappeared when the sintering temperature was 1150 ℃. SiO_2 can react with Al_2O_3 forming sillimanite ($Al_2O_3 \cdot SiO_2$). In this work, the final calcination temperature was 1200 ℃, and thus transformation from sillimanite into mullite occurred at 1175-1200 ℃, during which SiO_2 is engendered in the form of quartz strengthening the diffraction peaks of quartz. So, the diffraction peaks of quartz, corundum and mullite are detected in Fig. 5a.

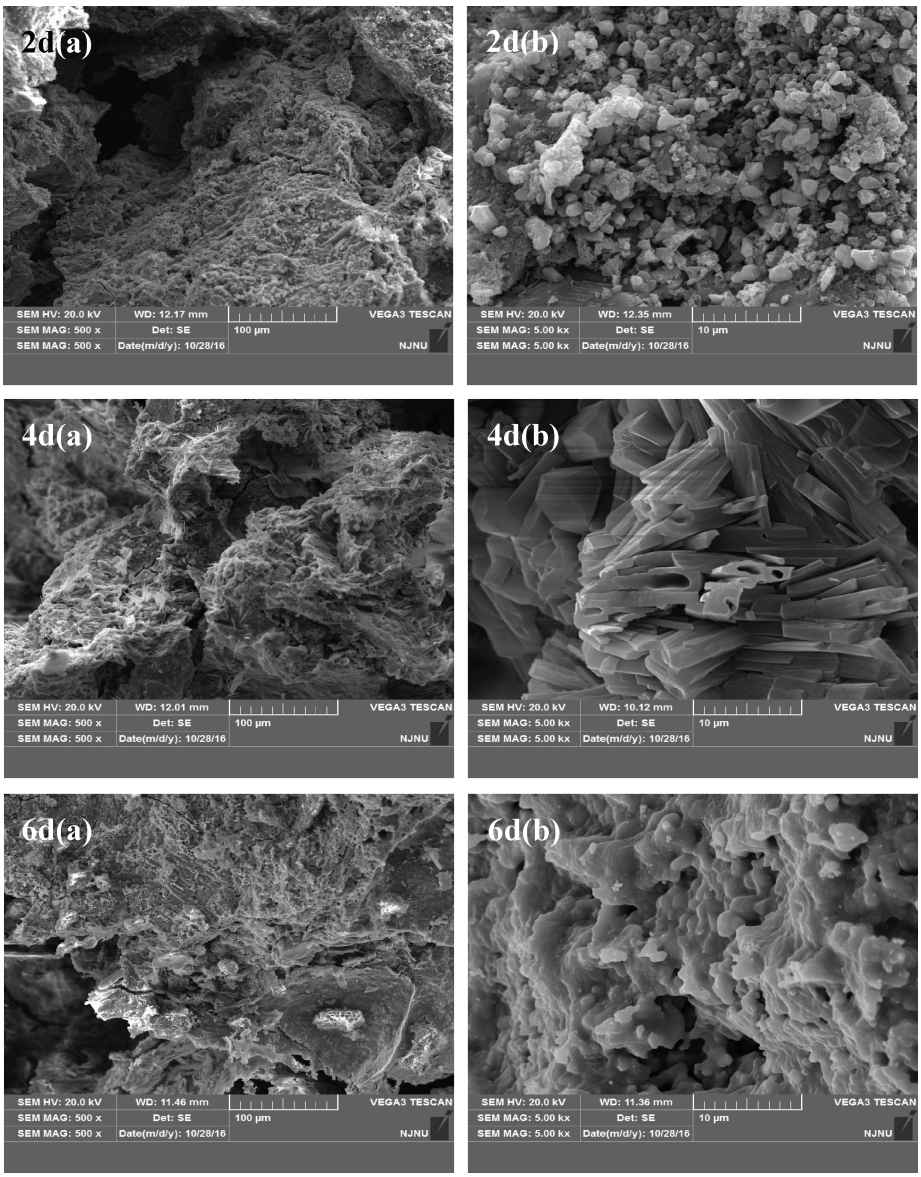

Fig. 4 SEM images of ceramic foams after sewage disposal

Cristobalite is chemically inert and completely neutral pH, which makes it generally does not undergo chemical changes and induction reaction in complex chemical systems. In addition, it has the advantages of high temperature and acid-alkali corrosion resistant which makes it is not deteriorated even at extremely high temperature or in harsh acid-alkali environment. Corundum has a

performance of high hardness and high melting point. Mullite is also chemically inert, and is a kind of high-quality refractory. The three main components make ceramic foams have good performances of acid-alkali corrosion and high temperature resistant, and thus ceramic foams maintain a relatively stable state in a variety of complex environments.

Fig. 5b is XRD pattern of ceramic foams supported Nickel catalysts by dipping ceramic foams into $NiCl_2$ solution for 6d and reacting with excess NaClO and NaOH. Compared with Fig. 5a, it can be seen that in addition to the diffraction peaks of mullite ($3Al_2O_3 \cdot 2SiO_2$), corundum (Al_2O_3), and quartz (SiO_2), the diffraction peak of NiOOH appears, which is due to the reaction between $NiCl_2$ and excess NaOH and NaClO solution. A strong diffraction peak of NaCl was also detected in XRD pattern, because the excess NaClO and NaOH are added to produce NaCl. The specific reaction is given Formula 1.

Fig. 5c shows the XRD pattern of ceramic foams after simulation of sewage treatment. Compared with Figs. 5a and 5b, there are still obvious diffraction peaks of mullite, corundum, and quartz, and also obvious diffraction peaks of NiOOH and NaCl. It can be seen that the main composition of the sample after simulated sewage treatment is similar to that of the specimen before sewage treatment, and NiOOH is still existed, which is consistent with the SEM analysis. Thus, NiOOH catalyst loaded on ceramic foams can be considered reusable.

3.4 Catalytic performance

In the single factor experiments, the benchmark of the temperature, dosages of sodium hypochlorite and catalyst, pH value are set as 35 ℃, 15.0 mL, 1.0 g and 8, respectively. The target value of each group of experiment is the total fading time of methylene blue, and the data are recorded in Tab. 4. It can be seen from Tab. 4 that the most obvious influencing factor is the reaction temperature.

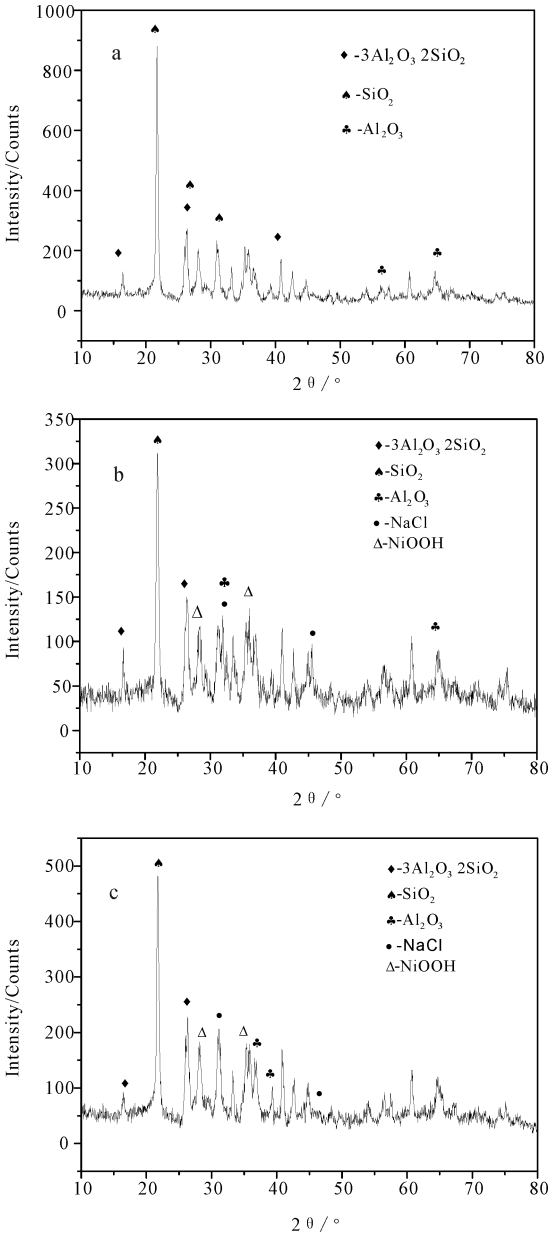

Fig. 5　XRD patterns of different specimens (a) Ceramic foams, (b) Dipping for 6d and reacting with NaClO and NaOH, and (c) After simulated sewage disposal

Tab. 4　The result of single factor experiment

No.	Catalyst dosage (g)	Temperature (℃)	pH value	NaClO dosage (mL)	Fading time (min)
1	1	15.0	8	15.0	2678
2	1	25	8	15.0	1158
3	1	35	8	15.0	386
4	1	45	8	15.0	200
5	1	55	8	15.0	131
6	1	35	3	15.0	514
7	1	35	5.4	15.0	426
8	1	35	10	15.0	367

(Continued)

No.	Catalyst dosage (g)	Temperature (℃)	pH value	NaClO dosage (mL)	Fading time (min)
9	1	35	12	15.0	334
10	0	35	8	15.0	523
11	0.5	35	8	15.0	485
12	1.5	35	8	15.0	356
13	2	35	8	15.0	343
14	1	35	8	5.0	590
15	1	35	8	10.0	454
16	1	35	8	20.0	374
17	1	35	8	25.0	351

The fading time of methylene blue shortened sharply with the increase of temperature, and the decreasing trend gradually slowed. At the same time, the fading time of methylene blue was also affected by the pH value, the dosages of catalyst and sodium hypochlorite. They were all negatively correlated, while the impact degree was nowhere near as the temperature. It only needed 131 min for the complete fading of methylene blue when the temperature, pH value, the dosages of catalyst and NaClO were 55 ℃, 8, 1.0 g, and 15.0 mL, respectively.

Nickel catalysts are mainly NiO, Ni_2O_3 and NiOOH. The catalytic mechanism of NiO can be explained as following. The crystal structure of NiO is NaCl type[21], oxygen can be transferred from the lattice to reactant molecules to form more than 2 species of nickel cations with variable valences. In addition, cations in the lattice can also be crossed soluble, forming a fairly intricate structure. This is because NiO belongs to the P type semiconductor, and nonstoichiometry can be caused due to the lack of positive ions, forming cation vacancies. In order to maintain the electroneutrality, two Ni^{++} transform into $Ni^{++(+)}$ near the hole. The latter can be regarded as a Ni^{++} bounded a hole "(+)"[22]. The hole transforms into a free hole with the increase of temperature, and can migrate on the solid surface and adsorb the negatively charged ions, such as ClO^-. At the same time, the hole can capture electrons of ClO^- and strip activated oxygen atoms to accelerate the decomposition of hypochlorite. In this work, Ni^{++} was hydrolyzed to Ni(OH) which was oxidized to NiOOH by sodium hypochlorite. The mechanism of the decomposition of sodium hypochlorite catalyzed by NiOOH catalyst in the system can still be explained by the redox catalysis reaction.

3.5 The optimal conditions for simulated sewage treatment

A catalyst is a substance that changes the speed of the chemical reaction of the reactants. However, it does not participate in the chemical reaction, and does not change the chemical equilibrium. NiOOH can be generated by the reaction between $NiCl_2$ and excessive mixed solution of NaOH and NaClO. The reaction does not happen, and methylene blue solution does not fade when only Nickel hydroxide is added into methylene blue solution at room temperature and 40 ℃ according to the literature[13]. However, the methylene blue can be oxidized to fade when only NaClO was added, while the fading time can run as long as 3 days. It can be concluded that NiOOH, as a catalyst, can be added to catalyze the chemical reaction between sodium hypochlorite and methylene blue. The speed changes of the reaction between methylene blue and sodium hypochlorite was explored in the presence of NiOOH catalyst.

Single factor experiments have proved that some factors have a certain influence on the fading speed of methylene blue. A further study on the optimal conditions for simulated wastewater treatment was carried out on the basis of single factor experiments. The optimum process parameters of sewage treatment under the action of Ni catalyst supported on ceramic foams was explored by an orthogonal test of four factors at three different levels. The temperature, pH, dosages of catalyst and sodium hypochlorite were selected as four factors, and the complete fading time of methylene blue was used as the target. The corresponding results were noted down, and the range analysis was also carried out, all shown in Tab. 5.

To some extent, the range reflects the discrete degree of the obtained data. The greater the

range, the influence of this factor on the fading time of methylene blue will be greater. Therefore, it can be concluded from Tab. 5 that the sequence of the influence factors on the fading of methylene blue is temperature > catalyst dosage > pH > sodium hypochlorite dosage according to the data in Tab. 5.

Tab. 5　The results of orthogonal test

Serial number	Temperature (℃)	Catalyst dosage (g)	pH value	NaClO dosage (mL)	Fading time (min)
1	25	0.5	5.4	10.0	1925
2	25	1	8	15.0	1421
3	25	1.5	10	20.0	1149
4	35	0.5	8	20.0	473
5	35	1	10	10.0	483
6	35	1.5	5.4	15.0	444
7	45	0.5	10	15.0	239
8	45	1	5.4	20.0	263
9	45	1.5	8	10.0	197
K_1	1498.33	879	877.33	868.33	
K_2	466.67	722.33	697	701.33	
K_3	233	596.67	623.67	628.33	
R	1265.33	282.33	253.66	240	

In a certain temperature range, with the increase of temperature, the fading time of methylene blue was dramatically decreased. The dosages of catalyst and sodium hypochlorite, and pH value also affect the reaction time, however, the influence degree was lesser as compared with the temperature. The reaction time was negatively related to all the factors mentioned above. As a whole, the higher the temperature, the greater the dosage of catalyst, the greater the pH value and the greater the dosage of sodium hypochlorite, the faster the reaction speed and the shorter the fading time of methylene blue are. The pH value of sodium hypochlorite itself is between 8-9, and the effect of pH value on the reaction time is not as obvious as before when the pH value is 8-9. The temperature has the most significant effect on the reaction time. In addition, the influence degree was more remarkable between 25～35 ℃ than 35～45 ℃ by comparing the effect of temperature on the reaction time. Taking into consideration the actual production cost, safety and maintenance of equipment, the reaction temperature, the dosages of catalyst and sodium hypochlorite can not be infinitely increasing. Therefore, the actual production and application conditions need to be taken into account when the series process parameters are increased. In summary, it can be concluded that the optimal process parameters of sewage disposal by ceramic foams supported Nickel catalyst are confirmed as that the temperature, pH value, and the catalyst and NaClO dosages are 35 ℃, 8, 1.0 g, and 15.0 mL, respectively.

4　Conclusions

Pores of the prepared foamed ceramic are interconnected with each other, with a large surface area and an open porosity of 43.53%. The main components of ceramic foams are mullite, cristobalite and alumina. The decomposition effect of sodium hypochlorite is the best, and the complete fading time of methylene blue is only 131 min when the temperature, pH value, the dosages of catalyst and NaClO are 55 ℃, 8, 1.0 g, and 15.0 mL, respectively. The sequence of significant influence factors on the fading of methylene blue is temperature > catalyst dosage > pH value > sodium hypochlorite dosage. Taking the practical application into comprehensive consideration, the optimal combination conditions for sewage treatment are confirmed as that the temperature, pH value, and the dosages of catalyst and NaClO are 35 ℃, 8, 1.0 g, and 15.0 mL, respectively.

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