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A Study on Kinetics of Decomposition of the Metal Oxides

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Abstract – Differential thermal analysis (DTA) and thermo-gravimetry (TG) of the individual oxalates and of mixtures of zinc oxalate with either nickel/iron oxalate or products of decomposition of the latter two, were carried out in air in sample cells made of different materials (Pt, Al, Al_2O_3, Ni). The information gathered from thermo analytical experiments, together with information derived from specific chemical tests for the evolution of carbon monoxide during decomposition, chemical analyses, XRD and stoichio-metric and thermo-chemical considerations helped to specify some of the inadequately explained features of the courses and kinetics of decomposition of the metal oxalates.

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INTRODUCTION

Zinc oxide presents the wurtzite structure and displays a high covalent Zn-O bond. ZnO is a wide band gap semiconductor extensively studied due to its intrinsic properties but with a limited industrial use as a UV-blocker in sun lotions as a component in mixed oxide varistors, as a catalyst/ photcatalyst. Additionally, forthcoming applications are envisaged as a gas sensor, solar cell and or / non-linear optical systems.

Of particular interest is the fact that ZnO can display novel nanostructures (nano-rings, nano-springs, nano-helices, and nano-bows), not typically observed in other oxides, due to the polar characteristics of their surface. Surface and quantum size effects have been described as responsible of compressive strain and band gap blue shift in nano-structured ZnO nano-particles.

However, surface effects and particularly, non-stiochiometry and the presence of hydrogen (forming part ort no of hydroxyls) seem persistent phenomena with larger influence in the oxide properties when comparing with quantum – alone. Although acoustic phonons are dominated by quantum confinement, optical phonons and visible (yellow/green) luminescence display properties mostly related to the presence of defects and/or hydrogen impurities without significant chemical/ physical sensibility to confinement.

Hubicki and Wojcik synthesized that platinum has been widely applied in catalytic industry and the recovery of noble metals from industrial wastes becomes an economic issue. The laboratory studies of platinum (IV) micro quantities removal from 1 M aluminium, copper, iron, nickel and zinc chloride solutions in 0.1 M hydrochloric acid solutions on the anion exchanger of the functional secondary and tertiary amine groups were carried out. For this anion exchanger the fraction extracted values (%E, Pt(IV)) as well as the sorption isotherms were determined depending on the king of aqueous phase and phase contact time.

Yadav and Kharkara followed liquid phase hydrogenation of a series of nitriles, namely, benzonitrile, butyrotirle, cinnamonitrile and crotonontrile over and has been investigated and the activities of the catalysts have been correlated with the structure of the catalysts on the basis of frontier orbital energy levels.

The as-synthesized samples and the materials derived from calcination and reduction were characterized by XRF, XRD, TGA, TEM, N_2 adsorption, H_2-TPR, XPS, and N_2O pulse chemisorption. Catalytic tests were carried out in a continuous flow-reactor at ambient pressure and 423–523K using H_2:C_3H_4 ratios of 1–12 and were complemented by operando DR IFTS experiments. The propyne conversion and propene selectivity correlated with the copper dispersion, which varied with the type of precursor or support and the calcination and reduction temperatures.

We examined the association of iron, copper and zinc between biological samples (scalp hair, whole blood and urine) and mortality from myocardial infraction (MI) patients of (first, second and third heart attack). We discoverd metal ions, especially with high chemical activity (e.g. redox-active Cu and Fe) must be carefully managed in biological systems. The “uncontrolled” activity, e.g. catalysis of Fenton-like reactions by ions like Cu(I) or Fe(II), is so damaging for the biological milieu that right from their entry, metal ions need to be strictly controlled until they arrive at their storage site.

The behaviour of Cu-ZnO catalysts in propene hydrogenation at 323 K has been investigated in order to gain information as to whether or not a synergic effect due to ZnO on the activity of copper is present. With this aim, two different series of catalysts were prepared by co-precipitation at (A) variable or (B) constant (≈ 8) pH. The whole composition range from CuO/ZnO 100:0 to 0:100 was covered in preparation A, while only copper-rich samples (CuO/ZnO≥67:33) were prepared from method B.

Chemisorption experiments of hydrogen and propene on samples reduced with hydrogen at 473 K point to the presence of adsorption sites in binary samples different from those existing in single components, that are influenced by the out-gassing temperature. The co-precipitated catalysts, which are already industrially employed, have higher catalytic activities than the kneaded ones, the optimum chemical composition being around Cu: Zn: Al = 60:35:5 (atom%) in both cases.

The total pore volume, the mean pore radius, and the porosity of the co-precipitated catalysts were three to four times larger than those of the kneaded ones. The pore-size distribution ranges from 50 to 5000 A for the co-precipitated catalysts and from 40 to 10,000 A for the kneaded ones. The former is equivalent to the copper and zinc oxide values in an individual state, while in the latter the values change in the composite form, with 80- and 20-A mean particle sizes, respectively.

The crystallite size of the composition with a higher catalytic activity after reduction was about 100 A both for zinc oxide and copper in the co-precipitated catalysts and 170–180 Å for copper and 270–280 A for zinc oxide in the kneaded ones. From these facts it was proposed that a finely mixed state in the oxidized precursor has its origin during the co-precipitation process.

Preparation of macro-porous bead adsorbents based on poly (Vinyl alcohol) Chitosan and their adsorption properties for heavy metals from aqueous solution were synthesized. A novel macro-porous bead adsorbents based on poly(vinyl alcohol)/chitosan (PVA/CS beads) were prepared, characterized and were used for the adsorption of heavy metal ions from aqueous solution.

The resulting PVA/CS beads were perfectly spherical in shape and exhibited good mechanical strength and chemical stability. The presence of NaNO3 (0– 0.137 mol/L) had little effect on Cu^{2+} adsorption, but the adsorption of Pb^{2+}, Zn^{2+} and Cd^{2+} decreased significantly in the same conditions. Various thermodynamic parameters were calculated and the results showed that the adsorption of all metal ions onto PVA/CS beads was feasible and endothermic in nature.

The results from the sequential adsorption– desorption cycles showed that PVA/CS bead adsorbents held good desorption and reusability, which would be a potential application in the fixed-bed continuous-flow column for the removal of heavy metals.

REVIEW OF RELATED LITERATURE

Kustov *et al.* worked on vanadia, copper and iron oxide catalysts supported on conventional $TiO_{2,}$ ZrO_2 and sulphated – TiO_2 and ZrO_2. These catalysts were characterized by elemental analysis, N_2-BET, XRD, and NH_3-TPD methods. The influence of potassium oxide additives on the acidity and activity in NO selective catalytic reduction (SCR) with ammonia was studied. The absolute activity of the samples does not vary significantly depending on the nature of the active metal and the acidic properties of the support used, seem to be influenced mainly by the concentration of active metal.

Zhang *et al.* produced the metal–silica interaction and cata lytic behavior of Cu-promoted Fe–Mn– K/SiO_2 catalysts investigated by temperature-programmed reduction/desorption (TPR/TPD), differential thermo-gravimetric analysis, in situ diffuse reflectance infrared Fourier transform analysis, and mossbauer spectroscopy. The Fischer–T ropsch synthesis (FTS) performance of the catalysts with or without copper was studied in a slurry-phase continuously stirred tank reactor. The characterization results indicate that several kinds of metal oxide–silica interactions are present on Fe–Mn–K/SiO_2 catalysts with or without copper, which include iron–silica, copper–silica, and potassium–silica interactions.

Komatsu and coworkers find out the catalytic activity of pure, doped nickel oxide, and mixtures of nickel oxides with different dopents were investigated by the reaction of carbon monoxide oxidation. The incorporation of lithium ions in the oxide enhanced the activity and the addition of indium lowered the activity. The activity of mixtures increased to several times greater than would be predicted by simple additive effect of single doped catalysts.

Deraz *et al.* synthesized alumina-supported NiO catalysts, promoted with 0.14-3 wt.% ZnO prepared by impregnation and then calcined at 400, 600, and 800 °C for 4 and 40 h. The phase analysis, surface and catalytic properties were investigated by using XRD technique, nitrogen adsorption at -196 °C, and oxidation of CO by O_2 at 200-300 °C ,respectively.

The results obtained reveal that ZnO doping of Ni/Al mixed oxides followed by calcination at 400 or 600°C for 4 h brought about slight increase in their specific surface area, which decreased progressively by increasing the calcination temperature of doped solids to 800 °C for 4 and 40 h. CO oxidation activity over NiO/Al_2O_3 mixed solids increased by treatment with ZnO followed by heating at 400 or 600°C for 4 h, and then decreased by increasing the calcination temperature to 800 °C for 4 and 40 h.

Zhuang and coworkers used by a thermal gravimetric balance reactor and a temperature-programmed reaction technique, it was found that carbon deposition on cerium oxide-containing nickel catalysts was decreased in both the induction and the constant carbon growth periods. Meanwhile the catalysts maintained activity for the steam reforming reaction. Based on this and our previous research a model for the promoting effect of cerium oxide is proposed.

Aai *et al.* followed the results of a leaching kinetics study of spent nickel oxide catalyst with sulfuric acid. The effects of spent catalyst particle size, sulfuric acid concentration, and reaction temperature on Ni extraction rate were determined. The results obtained show that extraction of about 94% is achieved using −200+270 mesh spent catalyst particle size at a reaction temperature of 85 °C for 150 min reaction time with 50% sulfuric acid concentration. The solid/liquid ratio was maintained constant at 1:20 g/ml. The leaching kinetics indicate that chemical reaction at the surface of the particles is the rate-controlling process during the reaction. The activation energy was determined as about 9.8 kcal/mol, which is characteristic for a surface-controlled process.

Zoo *et al.* studied the pre-reforming of commercial liquefied petroleum gas (LPG) over $Ni–CeO_2$ catalysts at low steam to carbon (S/C) molar ratios less than 1.0. It was found that the catalytic activity and selectivity depends strongly on the nature of the support and the interaction between Ni and CeO_2. The $Ni–CeO_2/Al_2O_3$ catalysts, which were prepared by impregnating boehmite (AlOOH) with an aqueous solution of cerium and nickel nitrates, exhibited the optimal catalytic activity and remarkable stability for the steam reforming of LPG in the temperature range of 275–375 °C. The effects of CeO_2 loading, reaction temperature and S/C ratio on the catalytic behavior of the $Ni–CeO_2/Al_2O_3$ catalysts were discussed in detail.

Mucka *et al.* showed the catalytic activities and some physico-chemical properties of NiO-ZnO catalysts prepared by precipitation of mixtures of carbonates and nitrates of both metals and subsequent calcination have been tested before and after irradiation, using the decomposition of hydrogen peroxide as a test reaction. A mutual influence of both oxides and their sensitivities to ionizing radiation has been proved.

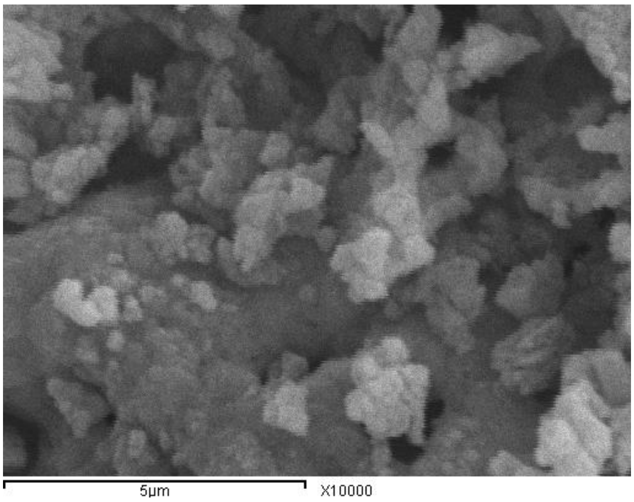
Busca *et al.* carried out the urea hydrolysis method to prepare well-crystallized Ni-Co-Zn-Al layered double hydroxides to be used as precursors of mixed oxide catalysts for the ethanol steam reforming (ESR) reaction. The calcination of the layered precursors gives rise to high surface area mixed oxides, being actually a mixture of a rock salt phase (NiO), a wurtzite phase (ZnO) and a spinel phase. The steam reforming of ethanol has been investigated over these catalysts after calcination at 973 K in flow reactor experiments. All these catalysts are active for ESR. At 820 K the selectivity to hydrogen increases with cobalt content. The most selective catalyst is the Ni-free Co-Zn-Al mixed oxide essentially constituted by a single spinel type phase $Zn_{0.55}Co_{0.45}[Al_{0.45}Co_{0.55}]_2O_4$.

Yang *et al.* reported the application of an inexpensive and easily-prepared lead oxide-manganese oxide catalyst combined with nafion (designated as Nf/PbMnOx) as a highly efficient air-cathode for a zinc-air battery. Mechanistic study of the reduction of O_2 for Nf/PbMnOx in alkaline aqueous solution using rotating ring/disk electrode voltammetry, and also an electrochemical approach using a wall-jet screen-printed ring disk electrode has been verified.

RESEARCH METHODOLOGY

Scandium – doped nickel copper oxide bulk, which consists of $Ni_{0.8}Cu_{0.2}O$, CuO, Sc_2O_3, and $Sc_2C_u2O_5$, responds only to NO_2 (50-500ppm) among NOx gases. Thin films of the oxide are prepared by an r.f. magnetron sputtering method, and their NOx-sensing characteristics are studied. The disappearance of crystalline $Sc_2Cu_2O_5$ in the film might affect the sensing performance for NOx. chemical vapor deposition (CVD) on Al_2O_3 substrates and characterized by means of glancing incidence X-ray diffraction (GIXRD), secondary ion mass spectrometry (SIMS) and field emission scanning electron microscopy (FESEM).

The obtained results revealed good responses even at moderate operating temperatures, with characteristics directly dependent on the system composition and nano – organization.



CuO nano particles were synthesized in a facile way, and characterized by scanning electron microscopy, transmission electron microscopy, X-ray diffraction, X-ray photo electron spectroscopy, and thermo gravimetry. Using these CuO nano particles, CuO functionalized QCM resonators were fabricated and explored for HCN sensing. The current results would provide an exciting alternative to fast, sensitive and selective detection of trace HCN, which would be of particular benefit in the area of public security and environmental application.

A novel room temperature solid state senor for the detection of hydrogen sulfide is described. The sensor was fabricated by first depositing a thin film of tin oxide on to a glass substrate followed by surface functionalizing with monolayer protected copper nano clusters (MPCs) capped with different capping agents prepared as per the brust synthesis. The response time for all the samples is smaller than 2 min.

Wang *et al*. studied low temperature H_2O sensor based on copper oxide/ tin dioxide thick film. nano structured tin dioxide (SnO_2) powders were prepared by a sol-gel dialytic process and the doping of CuO on it was completed by a deposition precipitation method. The thick film sensors were fabricated from the CuO/ SnO_2 polycrystalline powders. Sensing behavior of the sensor was investigated with various gases including CO, H_2, NH_3 hexane, acetone, ethanol, methanol and H_2S in air. It might have promising applications in the future.

Pd (IV) doped CO oxide composite nanofibers (PCNFs) have been successfully fabricated via electrospinning and then employed to construct an amperometric non-enzymatic glucose sensor.

Metal / Mixed metal oxides have wide application as photocatalyst. We synthesized copper sulphide powder. Thin films were developed by doctor blade deposition of Cu_xS and Cu_xS/TiO_2 composites with photocatalytic properties. The powder and thin film properties were characterized in terms of: the Fourier transform infra-red (FTIR), the X-ray diffraction (XRD), UV–vis spectroscopy, atomic forc e microscopy (AFM).The semiconductors association and the films homogeneity limit the electron- hole recombination, resulting in good efficiency in dyes photo degradation even under visible light irradiation.

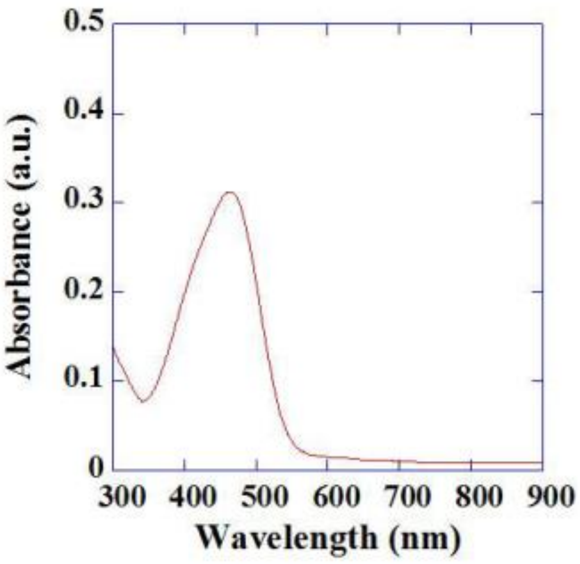
Simultaneous photo-reductive removal of copper (II) and selenium (IV) is studied for the first time using spherical binary oxide photocatalysts under visible light from a single contaminant, EDTA is found to be the best for Cu(II) reduction whereas formic acid is the best for Se(IV) reduction. In a mixed solution both EDTA and formic acid perform very well under visible light irradiation. Photo-deposited material is found to be copper selenide rather than pure copper and selenium metal. This indicates that the waste water containing copper and selenium ions can be efficiently treated under visible or solar light.

The study followed the advantage of copper doping into TiO_2 semiconductor photocatalyst for enhanced hydrogen generation under irradiation at the visible range of the electromagnetic spectrum.

Two methods of preparation for the copper-doped catalyst were selected – complex precipitation and wet impregnation methods – using copper nitrate trihydrate as the starting material. The dopant loading varied from 2 to 15%. Characterization of the photocatalysts was done by thermogravimetric analysis (TGA), temperature programmed reduction (TPR), diffuse reflectance UV-Vis (DR-UV-Vis), scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD).

DATA ANALYSIS

Iron-containing adsorbents prepared from methane fermentation sludge (MFS) were characterized by N_2 adsorption, XRD, SEM, EDX, pH determination and elemental analysis. The desorption studies were also performed and the mechanisms of Cu(II) adsorption was proposed the results indicated that the adsorbent obtained at 700^0C for 1 h in a steam atmosphere possessed the highest capability for Cu (II) adsorption. The invisibility of copper adsorption on the iron containing adsorbents is attributed to the formation of strong bonds between Cu (II) and the iron (hydr) oxides. The adsorbent can be applied to remove copper from water or soil by fixation onto the surface.



The research reviews the chemistry of Copper containing sulphur adsorbents in the presences of mere tans. A brief review of the chemistry of copper and thiols is given and a contrast is made to the behavior of copper-containing adorbents used for the desulfurization of liquid hydrocarbon streams that are routinely treated in refinery processes. At a temperature of approximately 150^0C very well formed lamellar crystals of copper (I) thiolate are formed, which indicates that the copper migreates from the surface of the catalyst to expose bulk copper for further reaction.

Chen–Shifu and coworkers developed p-n junction photocatalyst p-$CaFe_2O_4$/n-ZnO prepared by ball milling of ZnO in H_2O doped with p-type $CaFe_2O_4$. The Structural and optical properties of the p-n junction photocatalyst p-$CaFe_2O_4$/n-ZnO were characterized by X-ray powder diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), UV- Vis diffuse reflection spectrum (DRS) and fluorescence emission spectra . The mechanism of influence on the photocatalyst activity were also discussed by the p-n junction principle.

Smith *et al.* developed sulfated Fe_2O_3-TiO_2 (SFT) synthesized by treatment of immenite ore with sulfuric acid. The presence of sulfated Fe_2O_3-TiO_2 and mixed phase of Fe_2O_3- TiO_2 was confirmed by DRIFT spectra and XRD. The dispersion of sulfate displayed thermal stability up to 5000°C. The photocatalytic activity was evaluated by the oxidation of 4- chlorophenol (4-CP) in aqueous medium under UV-vis and visible light irritation.

The study induced Bi_2WO_6 photocatalyst synthesized via a facile low-temperature combustion synthesis method, using glycine as the fuel. The photocatalytic activities of the as-synthesized samples were evaluated by the photo degradation of rhodamine B (RhB) and phenol under visible – light irradiation. The photo catalyst showed much enhanced visible photocatalytic efficiency, up to 94.2% in 4 h, than the bulk Bi_2WO_6 powder (SSR) in the degradation of phenol.

The research examined a magnetically separable photocatalyst prepared by a continuous multi step spray pyrolysis process. In the first step, nickel ferrite core particles were prepared by an ultrasonic spray pyrolysis. In the second step, tetraethyl orthosilicate (TEOS) and titanium isopropoxide (TTIP) were sequentially injected and coated on the surface of the core particles coated layers were decomposed to form silica and tinania layers in a final furnace reactor. The titania- silica layered particles displayed higher photo activity.

The strong adhesion between the coated layer and the nickel ferrite core is attributed to the features of the multi-step process, where in the core particles are exposed to high temperature in the second reactor for only a few seconds and transformation of the core particles into non-magnetic particles is prohibited.

Liu and coworkers worked on the commonly used photocatalyst, TiO_2 (anatase) immobilized on porous nickel using 3 wt% polyvinyl alcohol (PVA) as the binder. The observance of photocatalytic degradation of Sal under pH values and initial concentrations was explained by the adsorption behavior of Sal. The parameters of the Langmuir Hinshelwood expression have been determined by different experimental ways.

Zhang *et al.* utilized titanium isopropoxide, ammonium carbonate and nickelous nitrate as the sources of titanium, nitrogen, and nickel to prepare titania photocatalyst co-doped with nitrogen and nickel by means of the modified sol–gel method. The effects of annealing temperature and component on the phase composition and photocatalytic activity were investigated. Nickel atoms existed in the form of Ni_2O_3, dispersed on the surface of TiO_2, suppressed the recombination of photo-induced electron-hole pairs, raised the photo quantum efficiency, and led to the enhancement of photocatalytic performance.

Shifu and coworkers chacterized p-n Junction photocatalyst NiO/TiO_2 was prepared by sol-gel method using $Ni(NO_3)_2.6H_2O$ and tetrabutyl titanate $[Ti(OC_4H_9)_4]$ as the raw materials. The results show that, for photocatalytic reduction of Cr_2O_7, the optimum percentage of doped-NiO is 0.5% (mole ratio of Ni/Ti). The photocatalytic activity of the p-n junction NiO/TiO_2 is much higher than that of TiO_2 on the photocatalytic reduction of Cr_2O_7. The mechanisms of influence on the photocatalytic activity were also discussed by the p-n junction principle.

CONCLUSION

Adsorption- based processes are widely used in the treatment of dilute metal – bearing wastewaters. The development of versatile, low-cost adsorbents is the subject of continuing interest. The effects of metal ion concentration, pH and contact time on binding ability was investigated by kinetic and equilibrium adsorption isotherm studies. The adsorption capacity for Pb (II), Cr (III), Zn (II) and Cu (II) was found to be 94.4 mg/g, 83.0 mg/g, 52.1 mg/g and 31.4 mg/g, respectively.

Wayne Turbeville and Nora Yap checked the chemistry of copper containing sulphur adsorbents in the presence of mercaptans. A brief review of the chemistry of copper and thiols is given and a contrast is made of the behavior of copper – containing adsorbents used for the desulfu rizatoin of liquid hydrocarbon streams that are routinely treated in refinery processes.

In addition, it is shown that bulk copper is involved in the adsorption of mercaptans, as there is significantly more sulfur than there is copper on the surface. Reaction mechanisms are proposed for the process of desulfurization of liquid hydrocarbon streams containing mercaptans when using a copper – containing adsorbent.

We analyzed the removal and preconcentration of Pb (II) from drinking tap water and wastewater samples via applications of newly modified three alumina physically loaded – dithizone adsorbents. Selective removal of Pb (II) from wastewater samples was accomplished with percentage recovery values of 94-99 + 1-2%, while the results collected from the selective preconcentration of Pb (II) from drinking tap water proved excellent percentage recovery values of 96-99+2-3% and 94-95 + 2-4% for the two studied concentration values 1.212 ng ml-1 and 4.800 g ml^{-1}, respectively.

In this study, characterization of adsorbent composition in co-removal of hexavalent chromium with copper precipitation is also analyzed. Mechanisms of hexavalent chromium co-removal with copper precipitation by dosing Na_2CO_3 were studied with a series of well-designed batch tests using solutions containing 150 mg l^{-1} Cu (II) and 60 mg l^{-1} Cr (VI). solubility products, neither copper-carbonate nor copper-hydroxide precipitates can be produced at pH around 5.0 for a pure 150 mg l^{-1} copper precipitation, characterization of copper-carbonate precipitates (adsorbent) was carried out through developing pC-pH curves of the systems by both equilibrium calculations and MINEQL^{+} 4.5 (a chemical equilibrium modeling software).

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