



PEOPLE'S DEMOCRATIC REPUBLIC OF ALGERIA
Ministry of Higher Education and Scientific Research
Ibn Khaldun University - Tiaret
Faculty of Material Sciences
Department of Chemistry



Major: Chemistry
Specialty: Materials Chemistry

**COURSE HANDOUT:
SYNTHESIS OF MATERIALS I**

Presented by:

Dr KEBIR-MEDJHOUDA Aouali Zohra

Foreword

This "Materials Synthesis I" practical work is intended for third-year bachelor's students, specializing in materials chemistry L3 CM. It is essentially based on the synthesis of different solid materials by developing their properties and applications.

This handout is divided into three parts, each part with a reminder that encompasses almost the entire target.

Chapter I will be devoted to practical sessions on the synthesis of metal complexes.

Then, in Chapter II, practical sessions will relate to different methods of synthesis of solids (sol-gel pathway, hydrothermal synthesis, synthesis by co-precipitation) and their physic-chemical analyses.





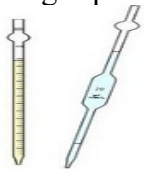



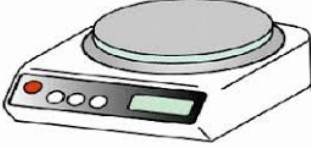

The last chapter deals with the part relating to the development of binders: definitions and classification, manufacture of plaster, preparation of cements for mineral additions.

Table of Contents

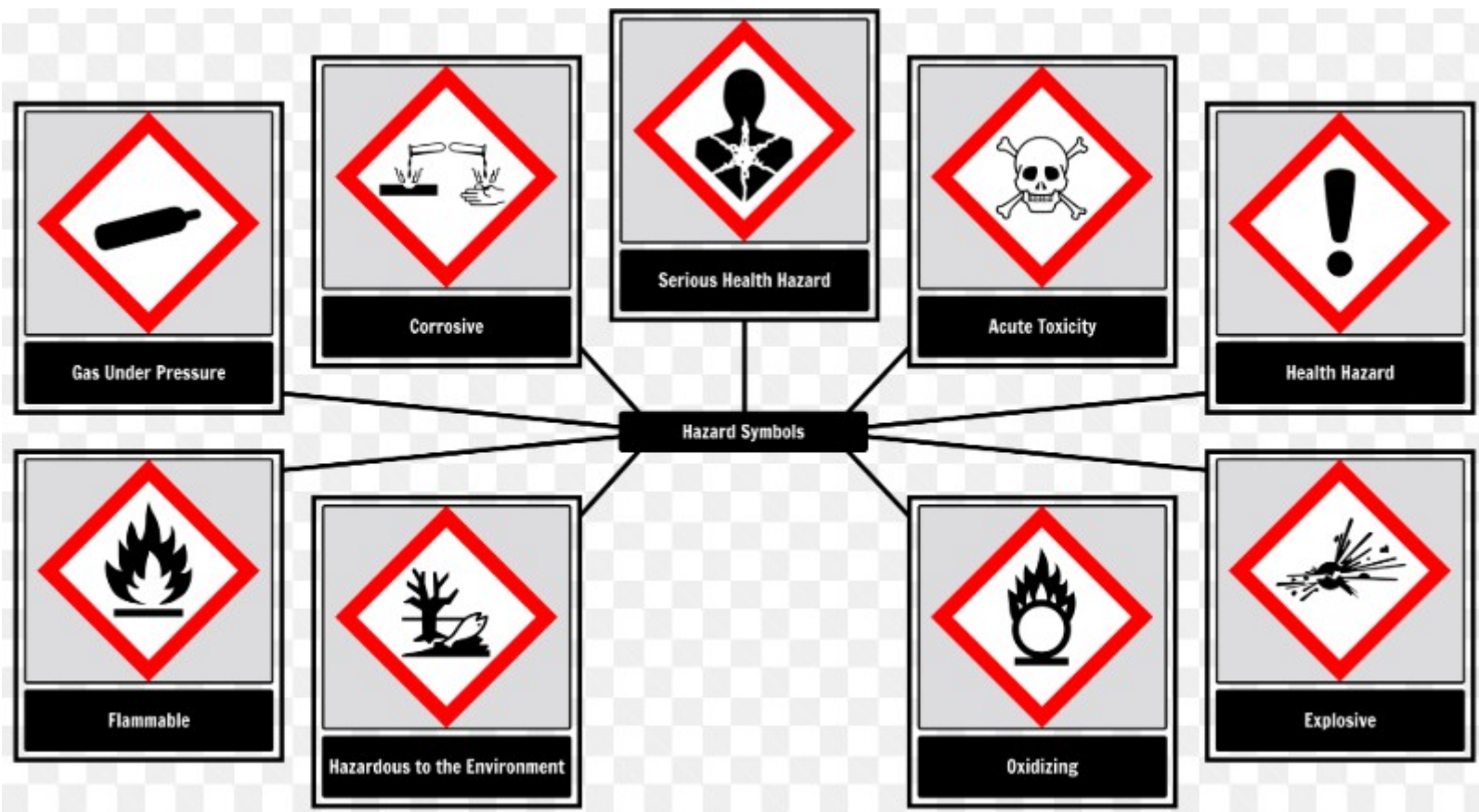
| | |
|--|----|
| Safety instructions..... | 1 |
| Pictograms..... | 2 |
| I. Synthesis of metal complexes..... | 3 |
| <i>PW 01: Synthesis of copper complex</i> | 5 |
| <i>PW 02: Synthesis of cobalt complexes</i> | 6 |
| II. Synthesis of solids..... | 7 |
| <i>PW 03: Synthesis of zinc oxide by the sol-gel route</i> | 9 |
| <i>PW 04: Synthesis of ferrite spinel of franklinite by co-precipitation</i> | 11 |
| <i>PW 05: Hydrothermal synthesis of zeolite</i> | 13 |
| III. Elaboration of binders..... | 14 |
| <i>PW 06: Making plaster</i> | 15 |
| <i>PW 07: Cement Manufacturing</i> | 17 |

Safety instructions

The 10 Safety Rules for Practical Work in the Lab

| | |
|--|---|
| <p>1 Wearing the gown</p>  | <p>2 Tying the hair</p>  |
| <p>3 Wash your hands regularly</p>  | <p>4 Not eating or drinking</p>  |
| <p>5 Using Pipettes</p>  | <p>6 Work in a fume hood as much as possible</p>  |
| <p>7 Use PPE (Personal Protective Equipment) if necessary: gloves, mask and goggles</p>  | <p>8 Organize the Workstation, Keep It Tidy, and Wash Glassware</p>  |
| <p>9 Use the right amount of product needed for handling</p> <p>balance</p>  | <p>10 Obey the waste disposal instructions</p> <p>Les déchets:</p> <ul style="list-style-type: none">→ Prévention et gestion des déchets→ Valorisation, élimination, réhabilitation, ...→ Suivi des flux de déchets→ et encore bien d'autres missions, ...  |

Pictograms



I. Synthesis of metal complexes

Introduction

The synthesis of metal complexes is a fundamental experiment in coordination chemistry. These compounds, where a central metal ion is surrounded by ligands (molecules or electron donor ions), have a wide variety of structures and properties, with many applications in catalysis, medicine and materials.

Targets of the practical work

- Master the techniques of synthesis of metal complexes.
- Characterize the complexes obtained by different methods (UV-visible spectroscopy, IR, NMR, etc.).
- To study the properties of these complexes.

Materials needed

- *Chemicals*: metal salts (CuSO₄, NiCl₂, etc.), ligands (ammonia, ethylenediamine, etc.), solvents (water, ethanol), precipitating agents (diethyl ether, etc.).
- *Glassware*: beakers, Erlenmeyer flasks, test tubes, pipettes, filters, etc.
- *Heating equipment*: hot plates, water baths.
- *Measuring equipment*: UV-visible spectrophotometer, IR spectrometer, etc.

Model Protocol

1. Preparation of the metal salt solution:

Weigh a precise amount of metal salt and dissolve it in a suitable solvent (usually water).

2. Ligand addition:

Slowly add the ligand to the metal salt solution under stirring.

Adjust the pH if necessary to promote the formation of the complex.

3. Isolation of the complex:

Precipitate the complex by evaporation of the solvent, by cooling or by the addition of a precipitating agent.

Strain and wash the precipitate with a suitable solvent.

4. Drying:

Dry the complex in an oven or sous vide.

Characterization of complexes

- UV-visible spectroscopy: Allows to study the electronic transitions in the complex and to determine its color.
- IR spectroscopy: Allows the identification of the characteristic vibrations of metal-ligand bonds and the ligands themselves.
- NMR: Allows the study of the chemical environment of atomic nuclei in the complex.
- Elemental analysis: Used to determine the elemental composition of the complex.

Examples of complexes to synthesize

- Copper complexes: $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4$, $[\text{Cu}(\text{en})_2]\text{SO}_4$
- Nickel complexes: $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$, $[\text{Ni}(\text{en})_3]\text{Cl}_2$
- Cobalt complexes: $[\text{Co}(\text{NH}_3)_6]\text{Cl}_3$, $[\text{Co}(\text{en})_3]\text{Cl}_3$

Precautions to take

- Wear protective eyewear and a lab coat.
- Handle chemicals with care, following safety instructions.
- Avoid contact with chemicals.
- In case of splashing into the eyes, rinse thoroughly with water and notify a teacher.

Conclusion

The synthesis of metal complexes is a rich and instructive experience that allows you to deepen your knowledge of coordination chemistry. By completing this lab, students will be able to develop their skills in handling, observation and interpretation of experimental results.

PW 01: Synthesis of copper complex

Target

The target of this lab is to synthesize metal complexes in order to determine part of the spectrochemical series of ligands, by synthesizing complexes having the same metal center at the same degree of oxidation but with different ligands.

Materials and reagents used

| Materials used | Reagents used |
|-----------------------------|---|
| Ice bath | Copper pentahydrate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ |
| Erlenmeyer flask | Ethanol |
| Filter equipment on Büchner | Concentrated ammonia |
| Filter paper | Ether |
| Spatula | |
| Watch glass | |

Handling

- Place 3.1 g of copper sulphate pentahydrate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in an Erlenmeyer flask.
- Add 3 mL of water and 5 mL of concentrated ammonia to the hood under the hood.
- Shake for 5 minutes (everything must be dissolved!), then add 5 mL of ethanol. Complex salt precipitates.

Remark:

It is also possible that some ligands are exchanged with ethanol, this phenomenon will not be taken into account.

- Place the Erlenmeyer flask in an ice bath for 40 minutes (still under the hood!).
- Wring out on Büchner. Wash the solid with 5 mL of concentrated ammonia, then 5 mL of ethanol and finally 5 mL of ether.
- Dry the solid as best as possible between two sheets of filter paper. Place the solid, well crushed and well spread, in a previously tapped Petri dish and dry it in the oven. When the solid is dry, note its mass.

Remark:

The dry character is determined by successive weighing: if the mass no longer varies, the solid is dry.

Questions

1. What is the color of the solution before and after ammonia is added?
2. Justifying this change in color
3. What is the precipitate formed when ammonia is added, give its chemical formula?
4. What happened to him, and why do we lower the temperature?
5. What happens when ethanol is added?
6. Why are crystals washed with diethyl ether and not with distilled water?
7. What is the effect of the nature of the ligand on the stability of the complex?
8. How does the geometry of the complex influence its properties?
9. What are the applications of metal complexes in catalysis and medicine?

PW 02: Synthesis of cobalt complexes

Target

The target of this lab is to synthesize metal complexes in order to determine part of the spectrochemical series of ligands, by synthesizing complexes having the same metal center at the same degree of oxidation but with different ligands.

Materials and reagents used

| Materials used | Reagents used |
|-----------------------------|----------------------------------|
| Ice bath | Cobalt (II) nitrate hexahydrate. |
| Erlenmeyer flask | Ethanol |
| Filter equipment on Büchner | Ammonia 6M |
| Filter paper | Ethylene diamine |
| Volumetric flask | Hydrochloric acid |
| Spatula | Ether |
| Watch glass | |

Handling

You will study the three ligands: H₂O, ethylene diamine, and ammonia with cobalt (II) as the metal center of the three desired complexes.

Handling 01:

Weigh 0.29 g of solid cobalt (II) nitrate hexahydrate (pink in color) and dissolve in 100 mL of distilled water.

Handling 02:

Weigh 0.29 g of cobalt (II) nitrate hexahydrate solid, add 0.20 mL of ethylene diamine, and then dilute the mixture in 100 mL of distilled water.

Handling 03:

Weigh 0.29 g of cobalt (II) nitrate hexahydrate solid, to which 1 mL of a 6 M ammonia solution is added, and then dilute in 100 mL of distilled water. The solution becomes slightly cloudy due to the formation of a blue precipitate. The solution remains rosy, however.

In order to verify that the precipitate possibly corresponded to a complex containing NH₃ ligands, a few milliliters of a 37% HCl solution were added.

Using a UV-visible spectrophotometer, perform the absorbance spectra of the previous solutions obtained.

Questions

1. What color is the solution after adding water?
2. What is the complex obtained in *handling 01*? And say his name.
3. What is the color of the solution in *handling 02*?
4. What is the complex obtained in *handling 02*? And say his name.
5. Justify the color change.
6. What happens when HCl is added to *handling 3*?

- Using the UV-screw spectrophotometer, give the absorption bands of each solution.

II. Synthesis of solids

Introduction

This lab offers an in-depth exploration of solid material synthesis methods, with a focus on three major techniques: the sol-gel pathway, hydrothermal synthesis and co-precipitation. These methods make it possible to obtain materials with specific properties; structural, morphological, functional and find many applications in catalysis, electronics, biomaterials, etc.

Targets of the practical work

- Master the fundamentals of these three synthesis methods.
- Synthesize solid materials at laboratory scale.
- Characterize materials obtained by different analytical techniques.
- Understand the influence of synthetic parameters on material properties.

Materials needed

- Chemicals: metal precursors (alkoxides, salts), solvents (water, alcohols), complexing agents, etc.
- Glassware: beakers, Erlenmeyer flasks, autoclaves, filters, etc.
- Heating equipment: hot plates, ovens, autoclaves.
- Measuring equipment: pH meter, conductivity meter, UV-visible spectrophotometer, X-ray diffractometer, etc.

Typical protocols

1. Sol-to-gel route

- *Hydrolysis*: Metal alkoxide reacts with water to form a gel.
- *Polycondensation*: The gel becomes denser by the formation of bonds between the monomer units.
- *Drying*: The gel is dried to obtain a porous material.
- *Calcination*: The material is calcined at high temperatures to remove organic compounds and obtain the final product.

2. Hydrothermal synthesis

- *Preparation of the reaction mixture*: The precursors are dissolved in a solvent (usually water) and placed in an autoclave.
- *Heating*: The mixture is heated at high temperature and high pressure for several hours.
- *Cooling*: The system is cooled slowly to allow the product to crystallize.

3. Co-precipitation

1. Solution preparation: Metal precursors are dissolved in separate aqueous solutions.
2. Mixing: The two solutions are mixed under stirring to form a precipitate.
3. Washing: The precipitate is washed with distilled water to remove impurities.
4. Drying: The precipitate is dried in an oven or by freeze-drying.

Characterization of materials

- X-ray diffraction (XRD): Determination of the crystal structure and size of crystallites.
- Infrared (IR) spectroscopy: Identification of functional groups present in the material.
- Thermal analysis (ATG, DSC): Study of thermal stability and phase transformations.
- Specific Area Measurement (BET): Determination of the specific area and porosity.

Examples of materials

- *Metal oxides*: TiO₂, ZnO, Fe₂O₃
- *Zeolites*.
- *Porous materials*.
- *Composite materials*.

Conclusion

Solids synthesis methods, including sol-gel, hydrothermal and co-precipitation, play a crucial role in determining the physicochemical properties of materials. Method choices influence the structure, morphology, crystallinity and functional performance of materials. Hydrothermal and sol-gel treatments are often distinguished by their optimal results in terms of catalytic performance and nanoparticle durability.

PW 03: Synthesis of zinc oxide by the sol-gel route

Target

Preparation of zinc oxide-based catalysts by the sol-gel method.

Introduction

Among the various methods used for the synthesis of materials, the Sol-Gel process is particularly well suited for the manufacture of homogeneous materials, in the form of powders and films.

During a so-called "citrate" sol-gel synthesis, the molecular precursors contained in the starting solution (the Sol) polymerize according to various mechanisms and form a network of oxides (the Gel). A drying step followed by heat treatments removes the organic compounds to form the inorganic oxide material. The name Sol-Gel is a contraction of the terms "gelling solution".

The Sol-Gel reaction takes place in two stages: the synthesis of the "soil", then the formation of the "gel".

Sol-gel reaction mechanism

The sol-gel process makes it possible to manufacture an inorganic polymer by a succession of simple chemical reactions and has a temperature close to room temperature (20 to 150°C).

The precursor

The precursor is a chemical reagent that initiates the reaction. It is often an alcoholate (alkoxide with the formula $M(OR)_n$: where M is a metal, and R is an alkyl organic group C_nH_{n-1} or a metal salt.

The synthesis of the "Sol"

A soil is defined as a stable dispersion in a liquid of colloidal particles. The synthesis of a "soil" is done at room temperature by adding water to an acidulated or basic organic solution containing precursors. This is the hydrolysis reaction. Subsequently, this "soil" can be made to evolve through condensation reactions into a three-dimensional network with infinite viscosity, called a "gel".



The formation of the "Gel"

A gel is defined as a two-phase system in which the solvent molecules (water, alcohol) are trapped in a solid network. When the liquid is water, it is called an aquagel or hydrogel, if it is alcohol, it is called an alcogel.



Materials and reagents used

| Materials used | Reagents used |
|--|--|
| Beakers Magnetic bars, Burette Pipettes Thermometer. Spatula Watch glass | Copper nitrates ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) Iron nitrates ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) Citric acid $\text{C}_6\text{H}_8\text{O}_7$ |

Handling

- Weigh 2.5g of copper nitrates ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) in 50mL of distilled water
- Weigh 5g of iron nitrates ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) in 60mL of distilled water
- Weigh 2mg of citric acid $\text{C}_6\text{H}_8\text{O}_7$ in 25 mL of distilled water

Preparation of CuFe_2O_4

This method is based on citric acid as a complexing agent. This method has the advantage of producing very fine powders with high homogeneity.

Remark

The starting materials required for the synthesis of powders are copper nitrates $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and iron $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. Water distilled as a solvent and citric acid $\text{C}_6\text{H}_8\text{O}_7$ is the complexing agent.

- Each quantity of metal nitrates [$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$], [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] and citric acid $\text{C}_6\text{H}_8\text{O}_7$ are dissolved in distilled water volumes.
- After the products have completely dissolved, mix the nitrate solutions in a single beaker which is placed in a water bath until the temperature is between 80 and 90 °C, with limited stirring.
- When heating, add the citric acid dissolved in the distilled water, drop by drop with the burette. The mixture remains under agitation in a water bath at a constant temperature (to evaporate the water) until a gel is obtained.
- Dry at 130 °C for 3 hours, then grind and calcine in an oven at 800 °C for 3 hours.

Questions

1. What is the impact of pH, temperature and reaction time on the properties of the materials obtained?
2. How do you choose the most suitable synthesis method to obtain a given material?
3. What are the potential applications of synthesized materials?

PW 04: Synthesis of ferrite spinel of franklinite by co-precipitation

Target

Synthesis of spinel ferrite of franklinite by the co-precipitation method.

Introduction

Spinel is a colored rock that appears in metamorphic rocks poor in quartz (gabbro, basalts, peridotites, etc.) but also in limestone rocks. When red, spinel resembles ruby, but does not have its commercial value.

Ferrites are used in the form of ceramics. Mixtures of iron oxide powders and bivalent oxides are carefully mixed and baked at high temperatures to form the ferrite by reaction in the solid state. It is then finely re-crushed, shaped with a pressed binder in the form of a bar, torus or wafer at high temperature (1200 to 1350°C) in a controlled atmosphere. These ferrites are "soft" magnetic, as they have three essential properties for applications:

- They have a very high resistivity, they are insulators except for Fe_3O_4 at high frequency.
- They have a very wide variety of magnetic properties.
- They have very low coercivity.

Co-precipitation

Is one of the simplest synthesis methods to implement, it consists of simultaneously precipitating at least two metal components in a solution. The precipitate obtained is washed, filtered, dried and then calcined to obtain mixed oxides. It goes through two stages:

1. Hydroxide co-precipitation

This technique consists of co-precipitating precursors in the aqueous phase, metallic salts, by the action of a base. The precipitates are of the form: $\text{M}_1\text{M}_2(\text{OH})_x, z\text{H}_2\text{O}$.

2. Heat treatment

Consists of removing water, passing through calcination between (700 °C to 1200 °C).

Materials and reagents used

| Materials used | Reagents used |
|-----------------------------|--|
| Beakers | Zinc chloride (ZnCl_2) |
| Magnetic bars, | Distilled water |
| Burette | Ferric iron chloride hexahydrate |
| Pipettes | ($\text{FeCl}_3, 6\text{H}_2\text{O}$) |
| Vials | Sodium Hydroxide Solution 3M |
| Thermometer. | |
| Spatula | |
| Watch glass | |
| Filter equipment on Büchner | |

Handling

1) Preparation of solutions:

1. 3M Sodium Hydroxide Solution

To prepare a sodium hydroxide (3M) solution in a 100 mL flask, one must weigh a mass $x(g)$ of soda and pour it into a 100 mL flask, add a little distilled water until the solid is completely miscibility and then continue to fill the water to the gauge line.

2. Zinc chloride ($ZnCl_2$) solution 25 mL

To prepare a zinc chloride ($ZnCl_2$) solution in a 25mL vial, weigh 1g of $ZnCl_2$ in a 25mL vial, add a little distilled water until the solid is completely miscibility and then continue to fill the water to the gauge line.

3. Ferric chloride ($FeCl_3$) solution 25mL:

To prepare a solution of iron chloride ($FeCl_3$) in a 25mL vial, weigh 1g of $FeCl_3$ in a 25mL vial, add a little distilled water until the solid is completely miscibility and then continue to fill the water to the gauge line.

2) Preparation of $ZnFe_2O_4$ spinel ferrite (franklinite)

In a 250 mL beaker, introduce 25 mL of the ferric chloride solution and 25 mL of the zinc chloride solution, add 20 mL of distilled water, stir magnetically and heat for 15 minutes at $80^\circ C$.

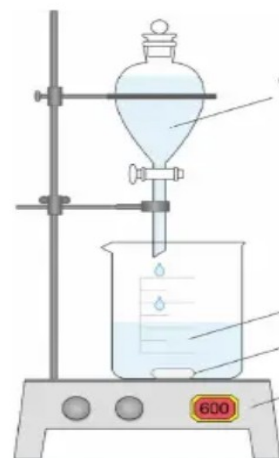
Put the sodium hydroxide solution in a burette and start dripping the solution using a titration setup with metric pH monitoring until a precipitate is formed according to the figure next to it.

Record the volume of NaOH as the mixture precipitates and its pH.

Filter and recover the precipitate on a previously weighed filter paper.

Dry in the oven at $105^\circ C$ until the mass is constant.

Calcine the final product at $700^\circ C$.



Questions

1. Calculate the mass (x) required to prepare a NaOH solution (3M).
2. What is the color and appearance of the precipitate?
3. To give the equilibrium of the reaction of formation of metal hydroxides.
4. To give the equilibrium of the ferrite formation reaction.
5. Analyze the sample obtained by XRD and IR
6. Draw by Origin, diffractogram and IR spectrum.
7. What are the results obtained?

PW 05: Hydrothermal synthesis of zeolite

Target

Hydrothermal synthesis of a porous aluminosilicate material of the zeolite type.

Introduction

A zeolite, is a microporous mineral belonging to the silicate group, a subgroup of tectosilicates. It has several properties such as ion exchange, adsorption and catalysis.

Materials and reagents used

| Materials used | Reagents used |
|---|---|
| Beakers Magnetic bars, Pipettes Agitator Spatula Watch glass Autoclave Filter equipment on Büchner | Silica Soda Sodium aluminate Distilled water |

Handling

In a 200mL beaker, mix 10g of silica source with 1g of aluminum source, then pour 12mL of water and put under magnetic stirring. Gently add 4g of the soda and shake for 1 hour. This is the stage of formation of the crystallization gel.

Put this gel in a stainless-steel autoclave, then in an oven at 90°C for 24 hours. After crystallization, the mixture is filtered, washed and then dried overnight at 60°C.

Weigh the product obtained.

Questions

1. Describe the properties and application of zeolite.
2. What is the color of the product obtained?
3. What is the material condition of the product obtained?
4. What is the role of the autoclave?
5. After weighing the final product, conclude.

III. Elaboration of binders

Definition of a binder

A binder is a product that is used to agglomerate a solid mass, solid particles in the form of powder or aggregates. Binders are used in the manufacture of concrete, mortars and even paints, glues, sealants, etc. etc.

Classification of binders

Depending on their composition, binders can be classified into two main families:

1. Mineral binders

Are generally obtained by high-temperature treatment of mineral matter, and are set in the presence of water. Carbon is only found in the oxidized form of CO_2 (carbonate), they can be classified into two families according to their hardening mode:

- a) *Aerial binders*: hardening in the air due to a carbonation reaction: aerial lime, plasters, clays, magnesium binders.

- b) *Hydraulic binders*: hardening in humid environments or in water due to a hydration reaction of silicates or aluminates: hydraulic lime, prompt cement, cements (Portland cement), slags.

2. Organic binders

Are synthesized by living organisms, or by human science, from mineral matter or pre-existing organic matter. Their method of setting is complex, and carbon is mainly found under the $\equiv\text{C}-\text{H}$ bond.

- a) *Hydrocarbon binders*: bitumen, tars.

- b) *Resins and especially polymers*: aminoplasts, for example, are polymers widely used as binders in the wood and wood products industry.

- c) *Oils*: are used for their drying power: linseed oil, ... etc.

PW 06: Making plaster

Target

Synthesize and shape the plaster.

Definition of plaster

It is mainly gypsum, gypsum deposits are numerous, particularly in France and the United States. They are mined in underground or open-pit quarries. Crude gypsum contains about 90% calcium sulphate, and for example, limestone CaCO_3 , magnesium carbonate MgCO_3 , clay and silica.

Plaster production

The manufacture of plaster from natural gypsum involves three steps:

- Extraction and preparation of gypsum.
- Cooking.
- Obtaining the finished products.

Materials and reagents used

| Materials used | Reagents used |
|--|---|
| Beakers Magnetic bars, Pipettes Vials Thermometer. Spatula Mortar Sieve Cups | Gypsum Distilled water Potassium sulphate solution at 0.1M. |

Handling

1. *Exothermicity of the cast setting:*

Step 1:

Take the rock from the natural gypsum and grind it with a mortar, then put the powder in a sieve so that it has the same dimensions. Weigh 200g of gypsum and put it in an oven at 150°C for 1 hour. After drying, the gypsum turns into plaster.

Put 50g of natural gypsum and 50g of plaster obtained in plastic cups, and add 20mL of water to each cup, and mix until homogenized. Leave to rest and then remove from the mold after 15 minutes.

Step 2:

In a 200 mL beaker, pour 80 mL of water. Pour in 100 g of plaster, dispersing it over the entire surface. Follow the temperature of the suspension with a thermometer without mixing, for about

2 minutes. Homogenize, then pour into a disposable container (plastic cup). Follow the temperature again as a function of time, while performing the following handling.

2. *Adding an adjuvant:*

Repeat the previous handling (Step 2) by replacing the water with a solution of potassium sulphate at 0.1M.

Questions

1. Give the chemical formula of the plaster as well as its chemical name.
2. Give the reaction of obtaining the plaster.
3. What is the initial and final color after drying?
4. Explain this change in color.
5. What is the mass of the product after drying?
6. Explain this loss of mass.
7. What is the state of matter of the mixtures obtained in the two cups?
8. Conclude.
9. What is the maximum temperature reached in the first handling (step 2)?
10. How long does it take for plaster to be considered hard?
11. What is the maximum temperature reached in the second handling?
12. How long does it take for plaster to be considered hard?
13. Conclude.
14. What is the role of the adjuvant?

PW 07: Cement Manufacturing

Target

Synthesis and implementation of cement and its properties.

Raw material

Clinker, the main constituent of a cement, is obtained from a mixture (called "cru") of 80% CaCO_3 limestone and 20% clay (silicoaluminate). Correctors, iron ore (Fe_2O_3), bauxite (Al_2O_3), sand (SiO_2) are added to achieve the desired composition. Limestone must be free of magnesium ($2\% < \text{content}$).

Materials and reagents used

| Materials used | Reagents used |
|----------------|-------------------|
| Beakers | Gypsum |
| Vials | Distilled water |
| pH Paper | Clay |
| Spatula | Limestone |
| Mortar | Milkman |
| Thermometer | Hydrochloric acid |
| Cups | |
| Crucible | |
| Test Tube | |

Handling

Weigh 75g of limestone, 25g of clay, (limestone and clay are extracted from the quarries, then crushed), grind them and then mix the two solids in a crucible to put it in a calcination kiln for 30 minutes at 750°C , this is the stage of clinker formation.

Once the mixture has cooled, add 5g of gypsum and 0.5g of slag. Mix everything together and grind again. This is the stage of cement formation.

Weigh 50 g of the cement obtained, add 25 g of water, to a 200 mL beaker. Mix until homogenized and transfer to a plastic cup or ice cube mold. Wash the beaker immediately.

Temperature change

Immerse the thermometer for about a minute to observe the rise in temperature. Clean it immediately.

Evolution of pH

Using pH paper, determine the pH during the setting.

Also take the pH of a little water added to the cement after setting.

Cement density measurement

Weigh a small block of cement and immerse it in a test tube filled with water. Measure volume variation.

Dissolution in an acidic medium

Dip a small piece of cement in hydrochloric acid. Shake gently.

Questions

1. What is a limestone, say its name and chemical formula?
2. Why should limestone be magnesium-free?
3. What is the pH of the water after taking it?
4. What is the mass obtained?
5. What is the volume obtained in (cm^3)?
6. What is the density in (g/cm^3)?
7. Note the observations about density.

Bibliography

Books

websites

scientific articles