May 17th, 2026

Department of Chemistry

Duration: 10:00-11:30

Academic Master 1 – Analytical Chemistry

Coefficient: 01 ; Credits: 01

Full name: …………………………………………..……..... Registration N°: ……………..….

Exam Answer Key

Section 1: Theoretical Reasoning Questions (05 Marks)

1. Nanotechnology (Surface-to-Volume Ratio)

- **Answer:** As particle size decreases to the nanoscale, the **surface-area-to-volume ratio** increases dramatically. Theoretically, this means a much higher percentage of the material's atoms are located on the surface rather than in the bulk. These surface atoms have "dangling bonds" and high surface energy, providing a vastly increased number of active sites available for chemical interaction with pollutants.

2. Sol-Gel Synthesis (Network Formation)

- **Answer:** A "sol" is a stable suspension of solid particles in a liquid, while a "gel" is a continuous three-dimensional solid network that traps the liquid phase. Theoretically, pH control is vital because it dictates the **rate of hydrolysis and condensation**.

3. FTIR Characterization (Molecular Fingerprinting)

- **Answer:** If the components are only physically mixed, the FTIR spectrum will simply be a superposition (sum) of the individual spectra of both materials. Theoretically, if a **chemical bond** (like a covalent or hydrogen bond) has formed, you will observe a "red shift" or "blue shift" (change in frequency) of existing peaks, or the appearance of entirely new absorption bands that represent the new linkage between the polymer and the mineral surface.

4. XRD (Crystallinity and Phase Identification)

- **Answer:** Crystalline materials have long-range order, which causes X-rays to diffract at specific angles, producing **sharp, well-defined peaks** (Bragg peaks). Amorphous materials (like many biopolymers) lack this order and scatter X-rays randomly. Theoretically, this results in a broad, rounded "hump" known as an **amorphous halo**. By observing the intensity and width of these peaks, a student can determine the degree of crystallinity of the composite.

5. SEM/EDX (Morphology and Elemental Mapping)

- **Answer:** The SEM provides the **topography** (physical shape), while the EDX provides the **elemental composition**. If the surface remains smooth but the metal is present, it theoretically suggests that the adsorption occurred via a **monolayer molecular mechanism** (like chelation or ion exchange) at the atomic level, rather than through the physical precipitation of metal crystals or "clumps" on the surface, which would have appeared as rough deposits in the SEM image.

Section 2: Materials & Experimental (10 Marks)

- *Answer:* Hydrogen peroxide (H_2O_2).
- *Answer:* 6 mol/L.
- *Answer:* 2.0 g.
- *Answer:* 40°C.
- *Answer:* 30 minutes.
- *Answer:* 60°C.
- *Answer:* Scanning Electron Microscopy (SEM).
- *Answer:* 18 MΩ.
- *Answer:* pH 7.
- *Answer:* 200 mesh.

Section 3: Results & Discussion

- *Answer:* Light yellow.
- *Answer:* 23.8 mg/g.
- *Answer:* 24.7 mg/g.
- *Answer:* Around 50 minutes.

- *Answer:* 50 mg/L.
- *Answer:* pH 7.
- *Answer:* pH 10.
- *Answer:* 110 mg.
- *Answer:* 231 nm.
- *Answer:* It increased (from 0.38 to 0.45).

Section 3: Advanced Reasoning and Analysis Questions (05 Marks)

1. Morphological Comparison

- **Answer:** The raw graphite shows a **tightly stacked, irregular structure**. In contrast, the intermediate shows a **loose, expanded structure** with significantly **increased layer spacing**. This indicates that the oxygen radicals successfully intercalated between the graphite layers, weakening the van der Waals forces. This physical expansion is essential because it proves the material is ready to be exfoliated into thin nanosheets.

2. Justification of the "Green" Experimental Design

- **Answer:** Traditional methods using $KMnO_4$ and concentrated H_2SO_4 often result in heavy metal (manganese) residues and risk of explosion due to the formation of Mn_2O_7. By using H_2O_2 as the **sole oxidant** in a **dilute acidic medium**, the researchers eliminated the explosive risk and the need for heavy metal cleanup, making the process safer, faster, and more environmentally friendly (Green Chemistry).

3. Interpreting the Adsorption Mechanism

- **Answer:** Unlike raw graphite, which is relatively inert, GO is covered with oxygen-containing functional groups. These groups provide active sites that can interact with positively charged Cd^{2+} ions through **electrostatic attraction** and **surface complexation/chelation**. The functional groups essentially act as "chemical hooks" that capture the metal ions from the water.

4. Analysis of Adsorption Kinetics

- **Answer:** Stopping at 10 minutes would lead to an incomplete removal of pollutants. At the start of the process, there are many **vacant active sites** available, leading to a rapid initial adsorption. However, as time progresses, these sites become occupied. By 10 minutes, the system has not yet reached the point where the rate of adsorption equals the rate of desorption (equilibrium), meaning the maximum removal capacity of the GO would not be reached.

5. Critical Thinking: The Role of pH in Heavy Metal Removal

- **Answer:** At a very low pH (pH 2), there is a high concentration of **hydrogen ions (H^+)** in the solution. These H^+ ions compete with the Cd^{2+} ions for the same active sites on the GO surface. As the pH increases to 7, the concentration of H^+ decreases, and the surface of the GO becomes more negatively charged (deprotonated), which reduces competition and increases the attractive forces for the Cd^{2+} ions.