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Structural Characterization and Development of an MXene/Cu-MOF Hybrid-Functionalized Quartz Crystal Microbalance for Gamma Radiation Sensing

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Abstract

MXene-based hybrid materials offer attractive electrical and surface properties for functionalizing quartz crystal microbalance (QCM) sensing platforms. In this study, an MXene/Cu-MOF composite was synthesized through a hydrothermal process and deposited onto a gold-coated quartz crystal to investigate its response to gamma irradiation. The functionalized QCM was exposed to a Cs-137 source, and its resonance frequency was monitored before, during, and after irradiation. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) were employed to examine the surface morphology and elemental composition of the composite before and after gamma exposure. The QCM response showed a progressive decrease in resonance frequency during irradiation. Relative to the pre-irradiation value, the frequency decreased by approximately 367 Hz at the end of the irradiation period and remained shifted during post-irradiation monitoring, reaching an overall decrease of approximately 768 Hz. Matched-magnification SEM images revealed irradiation-associated changes in agglomeration, particle packing, and surface heterogeneity. In contrast, the EDX results showed no substantial change in the detectable elemental composition of the analyzed regions, with only minor localized variations attributed to coating heterogeneity and substrate contribution. These findings demonstrate that the MXene/Cu-MOF-functionalized QCM exhibits a measurable and persistent frequency response to gamma exposure, accompanied by morphological reorganization of the sensing layer. The results support its potential as a proof-of-concept platform for monitoring irradiation-induced changes, while further studies involving control devices, replicate measurements, dose calibration, and complementary structural characterization are required to establish its gamma-sensing performance.

Introduction

The development of highly sensitive and selective sensors is of paramount importance for applications spanning environmental monitoring, industrial safety, and biomedical diagnostics. Among mass-sensitive transduction techniques, the Quartz Crystal Microbalance (QCM) has established itself as a versatile and highly sensitive analytical platform due to its real-time response, operational simplicity, and ability to detect nanogram-level mass variations [1]. The operating principle of QCM relies on the piezoelectric effect, wherein mechanical deformation of a quartz crystal generates an electrical signal. Building on this principle, Günter Sauerbrey demonstrated that the resonance frequency shift of the crystal is directly proportional to the mass change on its surface, forming the foundation of the Sauerbrey equation [2, 3]:

$$\Delta f = -\frac{2f_0^2}{A\sqrt{\rho_q \mu_q}}\Delta m$$

1

Where Δf is the frequency shift of quartz oscillator caused by a mass change Δm, ρ_q is the quartz density, μ_q is its shear modules, f_0 is the resonant frequency, and A is the active area of the crystal.

Despite its intrinsic sensitivity, the overall performance of a QCM sensor is predominantly dictated by the properties of the sensing layer, which governs analyte adsorption, selectivity, and signal transduction efficiency. Consequently, the design and engineering of advanced functional nanomaterials have become central to enhancing QCM-based sensing systems [4].

In recent years, two-dimensional (2D) nanomaterials have attracted significant attention due to their exceptional physicochemical properties. Among these, MXenes have emerged as a prominent class of 2D transition metal carbides and nitrides derived from MAX phases ($M_{n+1}AX_n$) via selective etching processes. These materials possess layered morphologies with surface terminations such as –OH, –O, and –F, which confer hydrophilicity and chemical versatility [5]. MXenes exhibit high electrical conductivity, large specific surface area, and excellent mechanical stability, making them highly attractive for sensing and energy-related applications [6, 7].

Simultaneously, Metal–Organic Frameworks (MOFs) have gained considerable recognition owing to their highly ordered porous structures, tunable surface chemistry, and exceptionally high surface areas. MOFs are constructed from metal ions or clusters coordinated with organic linkers, enabling precise control over pore architecture and functionality. In particular, copper-based MOFs (Cu-MOFs) are distinguished by their chemical stability, catalytic activity, and accessible metal sites, which facilitate strong adsorption interactions [8, 9]. The integration of MXene with Cu-MOF yields a hybrid nanostructure that combines the electrical conductivity and surface reactivity of MXenes with the porosity and adsorption capacity of MOFs. This synergistic combination is expected to significantly enhance mass loading, interfacial interactions, and overall sensing performance. However, systematic studies investigating MXene/Cu-MOF hybrid materials as functional coatings for QCM-based sensors remain scarce [10].

Radiation detection plays a crucial role in modern society due to the widespread use of ionizing radiation in medicine, nuclear energy, industry, environmental monitoring, and homeland security. Applications such as medical imaging, cancer radiotherapy, nuclear power generation, non-destructive testing, and radioactive waste monitoring require accurate and reliable radiation detection to ensure operational safety, protect human health, and minimize environmental risks [11–14]. Gamma-ray detection is also essential for emergency radiation monitoring, nuclear safeguards, and environmental surveillance.

Among the various types of ionizing radiation, gamma radiation is of particular importance because of its high penetration capability and photon-based nature. Gamma rays are electrically neutral, massless electromagnetic waves that travel at the speed of light and can penetrate dense materials such as metals and concrete. They are emitted during nuclear transitions, including radioactive decay, electron capture, and isomeric transitions, with energies typically ranging from 10^4 to 10^7 eV [11]. While these properties make gamma radiation indispensable in scientific, industrial, medical, and security applications, they also pose significant health and environmental risks. Consequently, the development of highly sensitive, reliable, and real-time gamma radiation sensors remains an important area of research.

In this context, the present work aims to develop and characterize a QCM sensor functionalized with an MXene/Cu-MOF hybrid layer for gamma radiation sensing. The structural, morphological, and elemental properties of the synthesized hybrid material were investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). In addition, the sensor performance under gamma irradiation was evaluated by monitoring changes in resonance frequency and adsorption behavior, providing insight into radiation-induced interactions at the sensor surface and the potential of MXene/Cu-MOF-based QCM sensors for gamma radiation sensing applications.

Experimental Details

2.1. Silicon Substrates Preparation

Silicone substrates (10 cm × 10 cm) were cleaned by immersion in ethanol for 10 minutes, followed by thorough rinsing with deionized (DI) water. Subsequently, the substrate surfaces were treated using Digital UV-ozone system (PSD Series, Novascan, USA) for 10 minutes. This treatment promotes surface activation through oxidative modification, thereby enhancing the adhesion of the deposited films.

2.2. Synthesis of MXene/Cu-MOF Composite

The MXene/Cu-MOF composite was synthesized using a hydrothermal approach. First, 180 mg of delaminated MXene was dispersed in 18 mL of deionized water and sonicated for 1 hour to obtain a uniform MXene suspension. Subsequently, 0.31 M copper nitrate trihydrate was added to this dispersion and stirred briefly to form solution 1.

In parallel, solution 2 was prepared by dissolving 0.17 M trimesic acid in 18 mL of ethanol. This solution was then gradually combined with solution 1 under continuous stirring to form a homogeneous mixture.

The resulting mixture was transferred into an autoclave and heated at 120°C for 12 hours. After the reaction, the autoclave was allowed to cool naturally to room temperature. The obtained blue precipitate was collected and repeatedly washed with ethanol to remove impurities. Finally, the product was dried at 60°C for 12 hours, yielding the MXene/Cu-MOF composite.

2.3. Preparation of MXene/Cu-MOF Solution

An MXene/Cu-MOF composite powder was dispersed in deionized water by adding 1.1 mg of the material to 1100 µL of DI water. The resulting suspension was ultrasonicated for 15 min to achieve uniform dispersion. Thereafter, 200 µL of the prepared solution was deposited onto the silicon substrate surface and allowed to rest for 24 h to ensure proper spreading and stabilization of the droplet prior to the drying process.

2.4. QCM Measurements

QCM measurements were performed using a QCM system (Fourien, Edmonton, Canada), which continuously monitors the resonance frequency of the coated crystal. For sensing experiments, the

MXene/Cu-MOF solution was deposited onto the gold electrode surface of the quartz crystal by drop-casting 20 µL of the suspension. The coated sensor was then allowed to stabilize for 10 minutes to ensure proper adhesion and equilibrium, during which frequency measurements were recorded.

To evaluate the sensor response to gamma radiation, the QCM chip was exposed to a Cesium-137 (Cs-137) source with an initial activity of 5 µCi and a half-life of 30.2 years (current activity: 2.95 µCi). The radiation source was positioned at a distance of 1.5 cm from the sensor, and the exposure was carried out for 1 hour at room temperature (25°C), as illustrated in Fig. 1. Afterward, frequency measurements were conducted for 1 hour following the removal of the source.

2.5. Samples Characterization

To evaluate the effects of gamma irradiation on the MXene/Cu-MOF samples, complementary structural and compositional characterization techniques were employed. Scanning Electron Microscopy (SEM) (JEOL JSM-7600F) was utilized to examine the surface morphology and microstructural features of the samples before and after irradiation. The SEM analysis was conducted at the King Abdullah Institute for Nanotechnology, King Saud University. The samples were exposed to gamma radiation for 3 hours at a fixed distance of 1.5 cm prior to post-irradiation imaging to assess any radiation-induced morphological changes.

In addition, Energy Dispersive X-ray Spectroscopy (EDX), integrated with the SEM system, was employed to determine the elemental composition and distribution of the samples. Comparative EDX analyses were performed before and after gamma exposure to identify any compositional variations or potential elemental redistribution induced by irradiation.

Results and Discussion

3.1 QCM Response

The QCM frequency response of the MXene/Cu-MOF sample pre-, during-, and post-gamma irradiation is shown in Fig. 2. QCM measurements revealed a gradual decrease in the resonant frequency of the MXene/Cu-MOF sample during gamma irradiation, and this decrease continued even after the irradiation had stopped. Based on the Sauerbrey relationship, which correlates a decrease in resonant frequency with an increase in mass loading on the quartz crystal surface [2], the observed frequency shift suggests radiation-induced changes in the composite, including enhanced adsorption and modified interfacial interactions between MXene and the Cu-MOF framework. The continued decrease after irradiation indicates long-lasting effects, possibly due to persistent structural modifications, ongoing adsorption, or slow stabilization processes within the composite.

Figure 3 presents the resonance frequency peaks of the MXene/Cu-MOF sample before, during, and after gamma irradiation. The resonance peaks shifted toward lower frequencies during irradiation and continued to shift after irradiation, suggesting persistent radiation-induced effects and ongoing changes

within the MXene/Cu-MOF composite. Compared with the resonance peak measured in air (without a sample on the QCM chip), a noticeable shift was observed after depositing the MXene/Cu-MOF sample onto the QCM chip. This shift indicates that the presence of the MXene/Cu-MOF coating alters the resonant behavior of the quartz crystal due to additional mass loading and changes in surface interactions. The resonance peaks shifted toward lower frequencies during irradiation and continued to shift after irradiation, suggesting persistent radiation-induced effects and ongoing changes within the MXene/Cu-MOF composite.

The resonance frequency values at each 10 min intervals, along with the corresponding frequency shifts and gamma irradiation dose values, are summarized in Table 1. These data provide a quantitative description of the time- and dose-dependent response of the MXene/Cu-MOF sample pre-, during-, and post- irradiation. Figure 4 shows the time-dependent frequency shift values of the MXene/Cu-MOF sample. A progressive increase in the frequency shift is observed during irradiation, indicating a continuous response of the system to the applied radiation dose. After irradiation, the frequency shift does not return to its initial baseline, suggesting that gamma exposure induces long-lasting changes in the composite, likely associated with persistent structural modifications, adsorption effects, or slow relaxation processes within the MXene/Cu-MOF framework. These findings are consistent with the study by Patil et al. [15], which demonstrated that gamma irradiation can induce persistent chemical and structural changes in $Ti_3C_2T_x$ MXene. Such radiation-induced modifications may contribute to the sustained frequency shifts observed in the QCM measurements.

Table 1
Resonance frequency values, frequency shifts, and corresponding gamma irradiation doses of the MXene/Cu-MOF sample measured at 10 min intervals.

Stage	Time (min)	Dose (μGy)	f (Hz)	$\Delta f = f - f_0$ (Hz)
Pre-gamma irradiation	5	0	f_o = 3994141.78114	0
	15	0	3992975.57033	−1166.21081
During-gamma irradiation	25	6.18	3993047.54975	−1094.23139
	35	12.37	3992991.14411	−1150.63703
	45	18.55	3992879.32585	−1262.45529
	55	24.73	3992729.34624	−1412.4349
	65	30.92	3992654.70072	−1487.08042
	75	37.10	3992608.61273	−1533.16841
Post-gamma irradiation	85	0	3992553.86758	−1587.91356
	95	0	3992438.59209	−1703.18905
	105	0	3992205.28039	−1936.50075
	115	0	3992220.61021	−1921.17093
	125	0	3992218.35372	−1923.42742
	135	0	3992207.93525	−1933.84589

3.2 SEM Analysis of MXene/Cu-MOF Before and After Gamma Irradiation

Scanning electron microscopy was employed to examine the morphological characteristics of the MXene/Cu-MOF composite before and after gamma irradiation. To enable a consistent comparison, representative micrographs obtained at identical magnifications of 5000× and 20000× are presented in Fig. 5.

Before gamma irradiation, the SEM image obtained at 5000× (Fig. 5a) reveals a heterogeneous morphology with irregular particulate aggregates and localized anisotropic surface features distributed across the sample. Regions with different particle densities and feature dimensions are evident, indicating non-uniform organization of the hybrid material. At 20000× (Fig. 5b), the surface is composed predominantly of closely packed particles with irregular sizes and shapes. These particles form compact agglomerated domains and produce a rough, heterogeneous surface texture.

Following gamma irradiation, noticeable changes in surface morphology are observed. At 5000× (Fig. 5c), the sample exhibits larger irregular agglomerates with layered and plate-like surface features. The localized anisotropic structures observed before irradiation become less distinguishable, while broader and more compact domains are evident. These differences indicate irradiation-associated reorganization of the surface morphology. At 20000× (Fig. 5d), the irradiated sample displays irregular particulate domains together with elongated lamellar features distributed within the surface structure. Compared with the pre-irradiated sample, the particle arrangement appears less uniform, and the boundaries between individual particulate features are less clearly defined. These observations suggest changes in particle organization, agglomeration, and local surface architecture following gamma exposure.

Ionizing radiation is known to induce structural modification, defect formation, and atomic rearrangement in nanomaterials through radiation–matter interactions [16]. Gamma irradiation has also been employed to modify MXene-based hybrid materials by promoting nanoparticle formation, interfacial interactions, and structural reorganization [17, 18]. However, because these studies involved different material systems and irradiation conditions, they provide only general support for the possibility of radiation-induced morphological modification and should not be considered direct evidence of the mechanisms occurring in the present MXene/Cu-MOF composite. The manuscript's references [16–18] concern ionizing-radiation modification of nanomaterials and irradiation-assisted preparation of MXene-based hybrids.

Overall, the matched-magnification SEM comparison indicates that gamma irradiation is associated with morphological reorganization of the MXene/Cu-MOF composite, including changes in agglomeration, particle packing, and surface heterogeneity. Nevertheless, SEM alone cannot establish the chemical or crystallographic mechanisms responsible for these changes. The observed morphological variations may influence the accessible surface area, interfacial interactions, and mass-loading behaviour of the sensing layer, thereby contributing to the QCM frequency response.

3.3 EDX Analysis of MXene/Cu-MOF Before and After Gamma Irradiation

Energy-dispersive X-ray spectroscopy was used to examine the elemental composition of representative regions of the MXene/Cu-MOF composite before and after gamma irradiation. The EDX spectra, analyzed regions, and semi-quantitative elemental compositions are presented in Fig. 6, with panels (a) and (b) representing the sample before gamma irradiation and panels (c) and (d) representing the irradiated sample.

Before irradiation, the region shown in Fig. 6a contains C, O, Si, and Cu at 29.63, 12.55, 51.63, and 6.19 wt%, respectively. After irradiation, the region shown in Fig. 6c contains 29.79 wt% C, 11.74 wt% O, 52.24 wt% Si, 5.62 wt% Cu, and 0.60 wt% Ti. The carbon and silicon contents remain nearly unchanged, while only minor variations are observed in the oxygen and copper contents. The low-intensity titanium signal detected in Fig. 6c is consistent with the Ti-containing MXene phase. However, because titanium was

detected at only 0.60 wt% and was not consistently identified in all analyzed regions, its appearance should be attributed cautiously to local variations in MXene distribution, coating thickness, signal intensity, or the detection limit of the EDX measurement rather than to gamma irradiation itself.

In the second analyzed region, Fig. 6b shows 15.35 wt% O, 76.01 wt% Si, and 8.63 wt% Cu before irradiation. Following irradiation, Fig. 6d shows 15.32 wt% O, 75.68 wt% Si, and 9.01 wt% Cu. The close agreement between these elemental percentages indicates that no pronounced compositional change occurred within the analyzed regions after gamma exposure. Carbon was not included in the quantitative analysis of Figs. 6b and 6d. Therefore, the elemental percentages in these panels were normalized only to the elements selected during spectrum processing and should not be compared directly with the complete elemental compositions reported in Figs. 6a and 6c. The high silicon content detected in all analyzed regions indicates that the electron interaction volume extended into the underlying silicon substrate. This observation is consistent with a relatively thin and/or locally non-uniform MXene/Cu-MOF coating. The detection of copper supports the presence of the Cu-MOF component, while carbon and oxygen are associated with the MXene/Cu-MOF composite and surface-containing species.

Overall, the EDX analysis confirms the presence of the principal elements associated with the MXene/Cu-MOF coating and the silicon substrate before and after gamma irradiation. No major change in elemental composition is evident from the analyzed regions. The small differences observed among the spectra are likely related to localized compositional heterogeneity, variations in coating thickness, and substrate contribution. Although the SEM results indicate irradiation-associated morphological reorganization, the EDX findings suggest that these morphological changes occurred without substantial alteration of the detectable elemental composition. Further characterization using X-ray photoelectron spectroscopy, Raman spectroscopy, Fourier-transform infrared spectroscopy, or X-ray diffraction would be required to determine whether gamma irradiation affected the oxidation states, chemical bonding, or crystallographic structure of the composite.

Conclusion

In this study, the influence of gamma irradiation on the structural and compositional properties of MXene/Cu-MOF hybrid materials was systematically investigated using Quartz Crystal Microbalance (QCM), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Spectroscopy (EDX) measurements. The results demonstrate that gamma radiation induces pronounced modifications in both surface morphology, elemental distribution, and sensor response behavior.

QCM measurements demonstrated a measurable shift in resonance frequency upon exposure to gamma radiation, confirming the sensitivity of the MXene/Cu-MOF-functionalized sensor to radiation-induced mass and surface interactions. The observed frequency variation reflects changes in adsorption behavior and possible radiation-induced modifications at the sensor interface, highlighting the effectiveness of the hybrid coating in enhancing mass sensitivity and signal response.

SEM analysis revealed significant structural evolution following irradiation, including partial degradation of the original morphology, the emergence of layered and network-like architectures, and the formation of nanoscale defects such as voids and microcracks. These changes indicate radiation-induced atomic rearrangement and restructuring within the hybrid material. Complementary EDX analysis confirmed variations in elemental composition, including slight oxygen depletion and redistribution of constituent elements, further supporting the occurrence of radiation and matter interactions.

Additionally, a noticeable change in the sample color was observed after irradiation, suggesting alterations in surface chemistry, potentially associated with oxidation processes or the formation of new chemical bonds. Such modifications are likely to influence the electronic, mechanical, and adsorption properties of the material.

Overall, the findings confirm that gamma irradiation can effectively tailor the structural and physicochemical properties of MXene/Cu-MOF hybrid materials. The combined QCM, SEM, and EDX results demonstrate that these radiation-induced modifications significantly influence sensor performance, particularly in terms of sensitivity, adsorption characteristics, and resonance frequency response. These outcomes highlight the strong potential of MXene/Cu-MOF-functionalized QCM sensors for advanced gamma radiation detection applications.

Declarations

Author Contribution

Conceptualization and supervision, A.N.A.; methodology, investigation, and data curation, N.A., R.A., S.A., and A.A.-M.; formal analysis and visualization, N.A. and A.N.A.; writing—original draft, N.A.; writing—review and editing, all authors. All authors read and approved the final manuscript.

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Figures

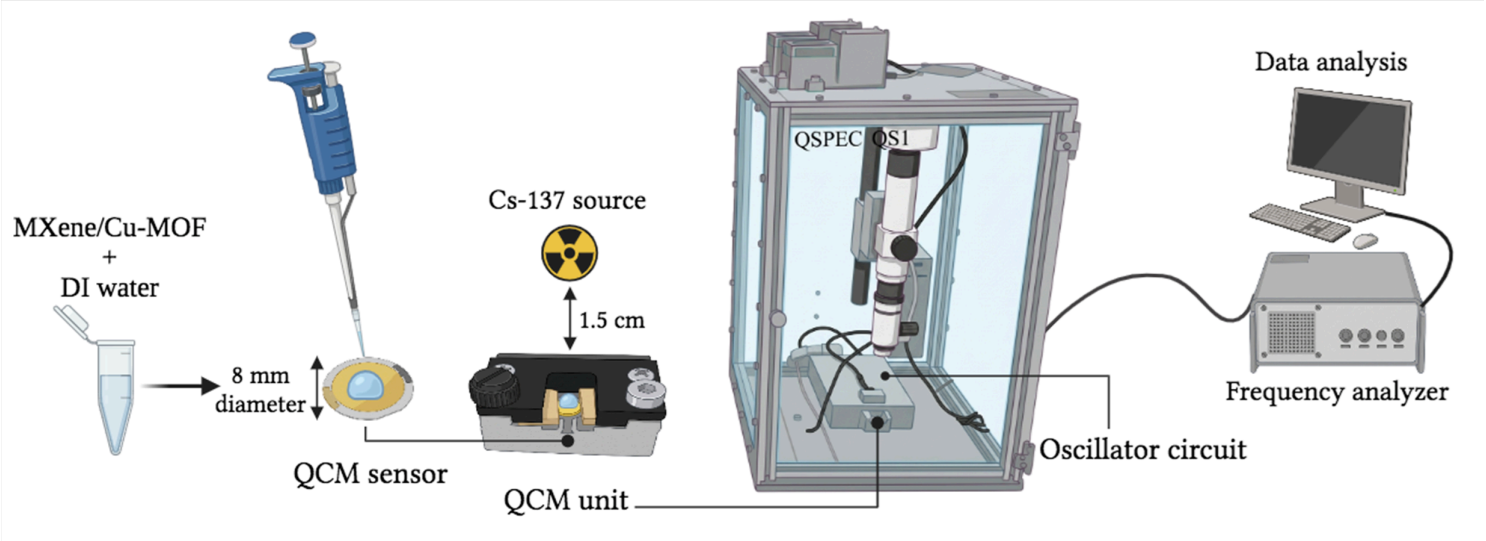


Figure 1

Schematic of an MXene/Cu-MOF-functionalized QCM sensor for gamma-ray detection.

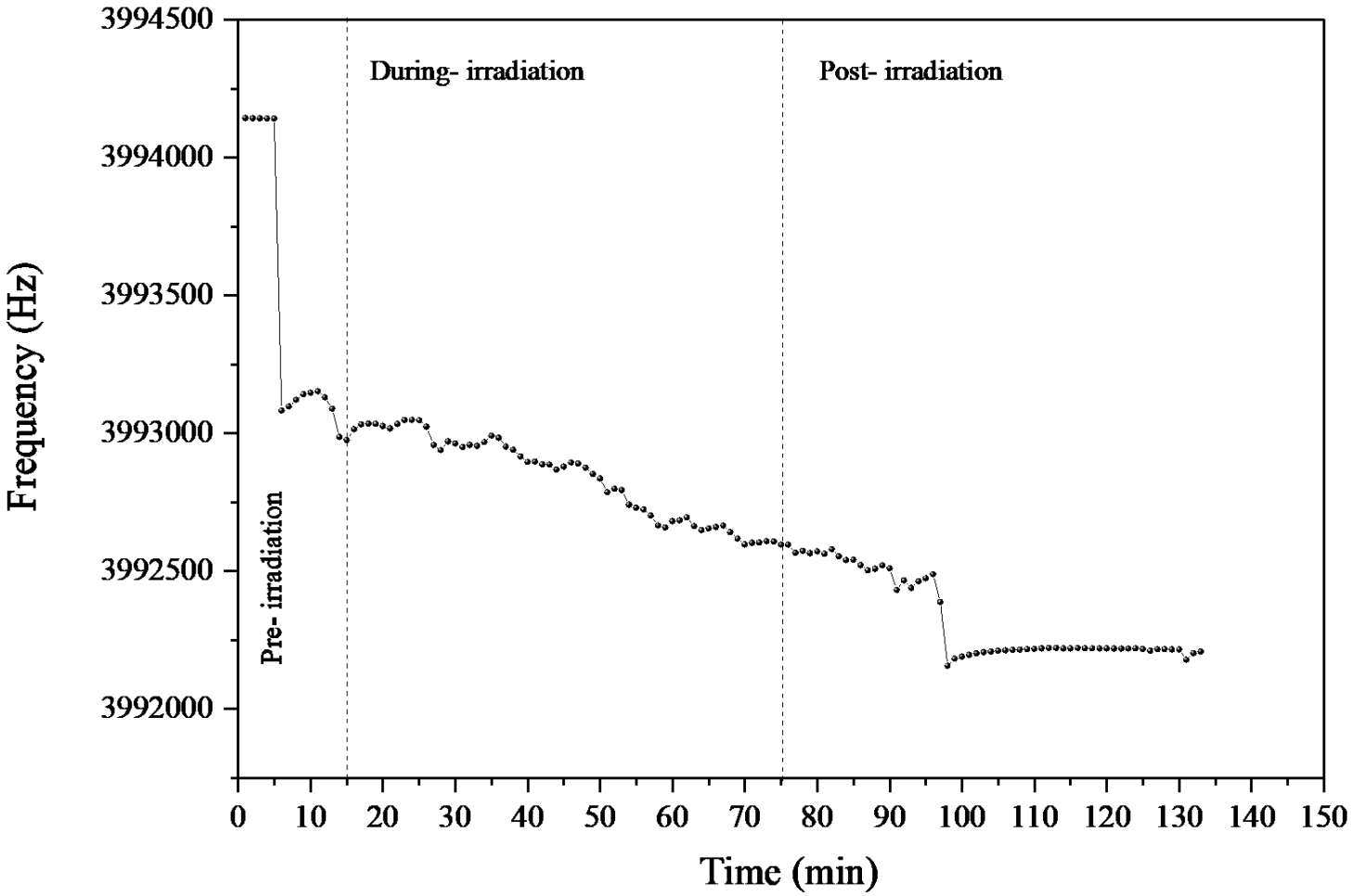


Figure 2

Frequency changes over time in the pre-irradiation, during-irradiation, and post-irradiation periods.

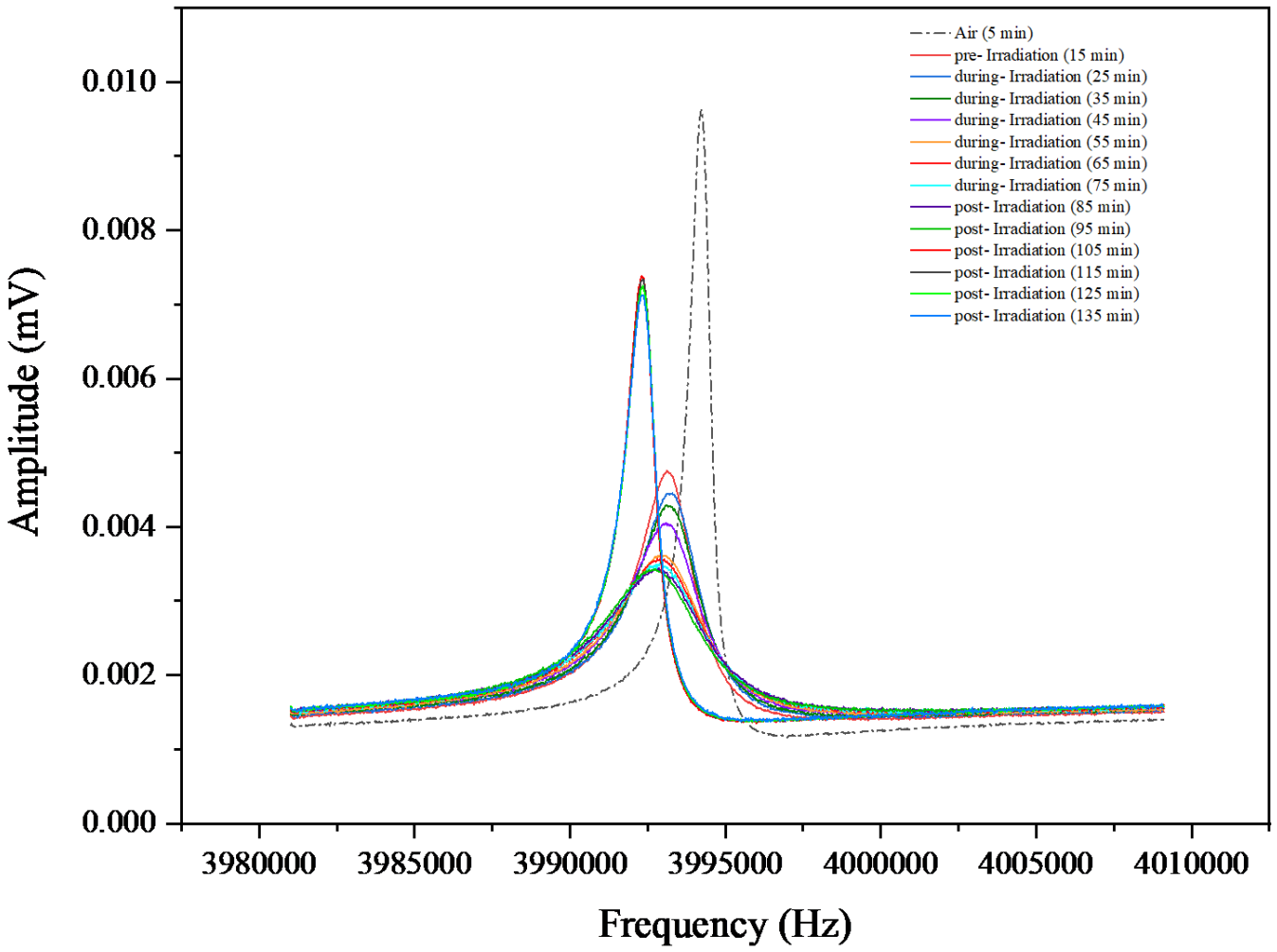


Figure 3

Resonance frequency peaks of the MXene/Cu-MOF sample pre-, during-, and post- gamma irradiation.

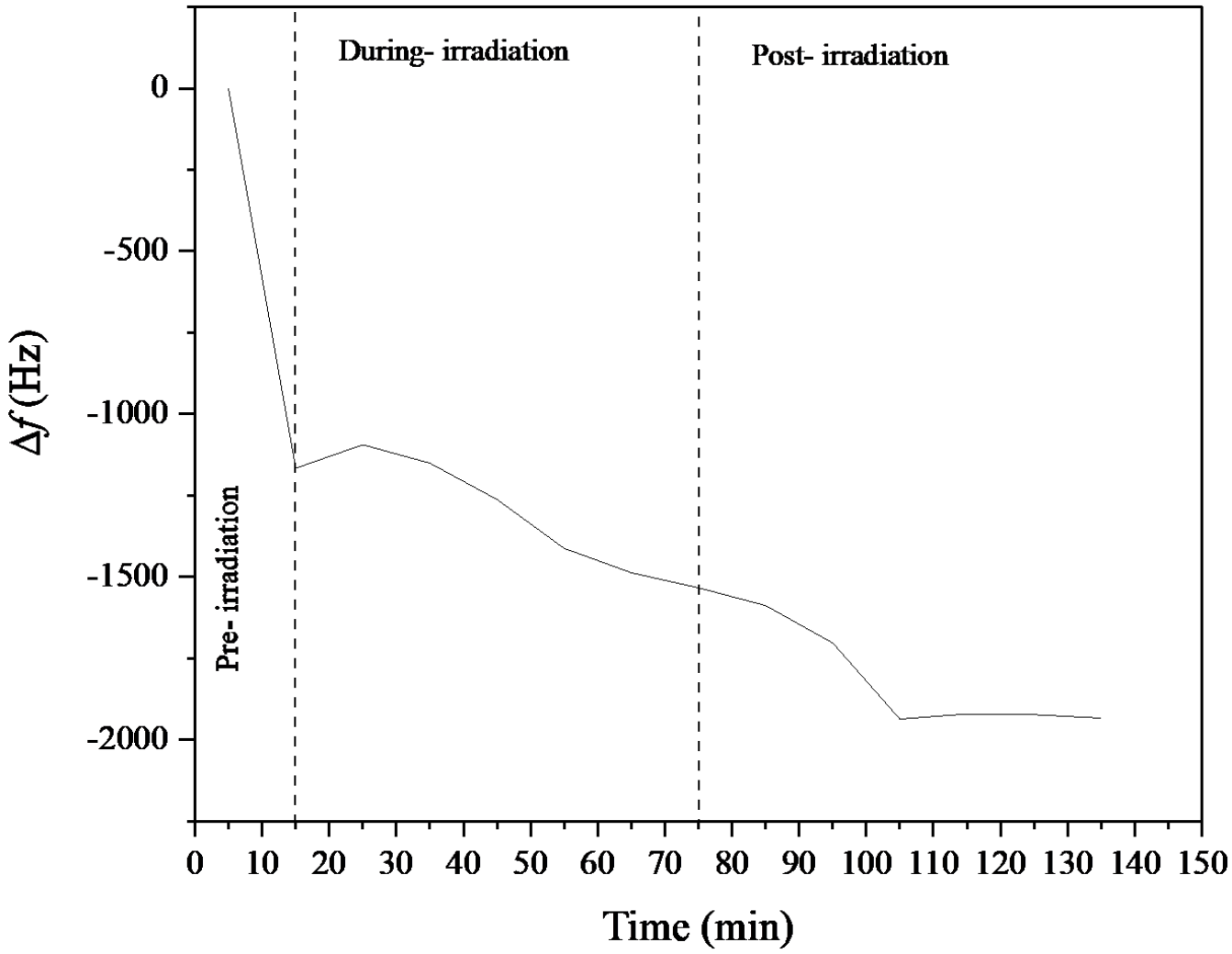


Figure 4

Frequency shift as a function of time for the MXene/Cu-MOF sample pre-, during-, and post gamma irradiation.

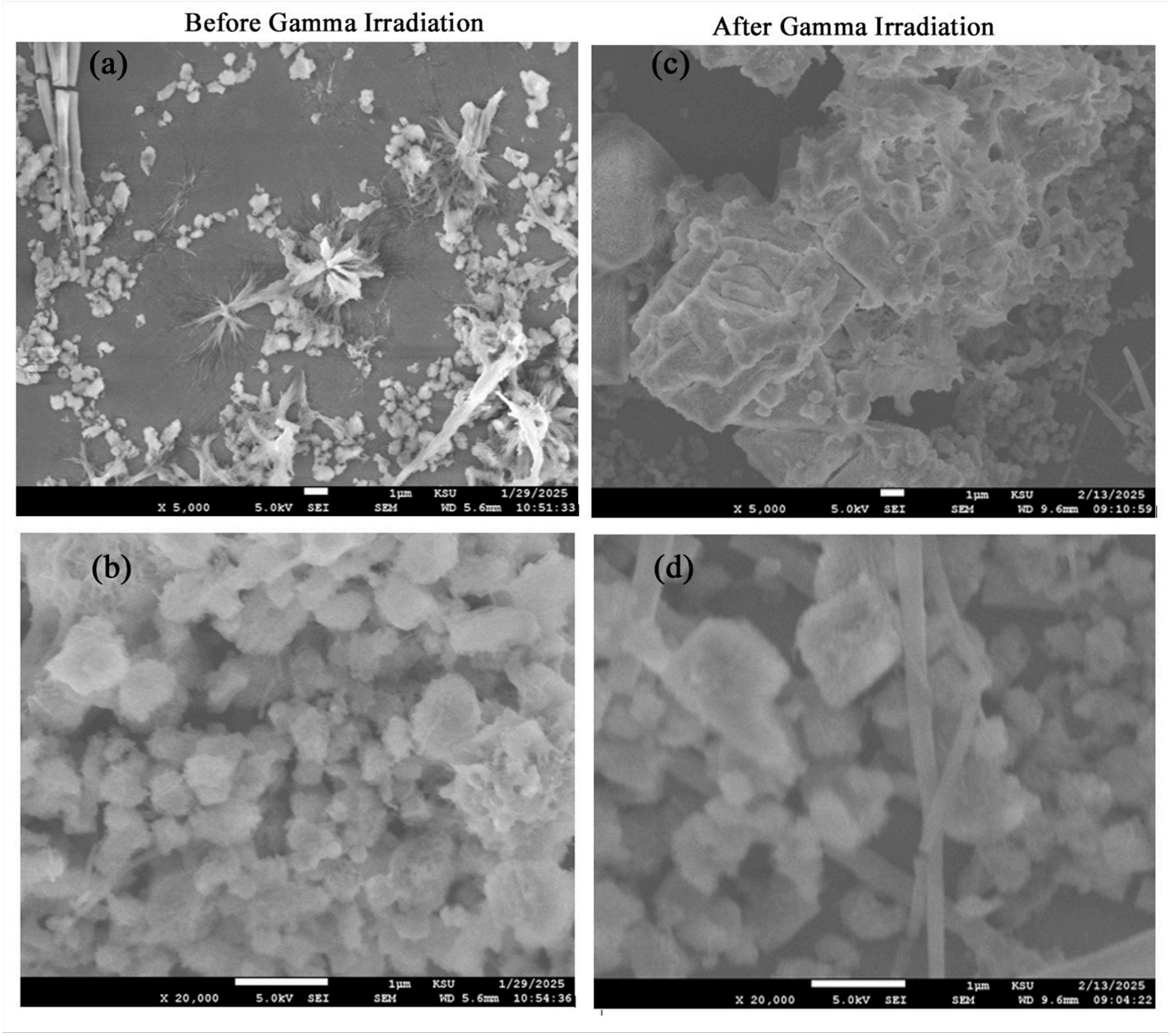


Figure 5

SEM micrographs of the MXene/Cu-MOF composite before gamma irradiation at two different magnifications: (a) 5000× and (b) 20000×, and after gamma irradiation at (c) 5000× and (d) 20000×.

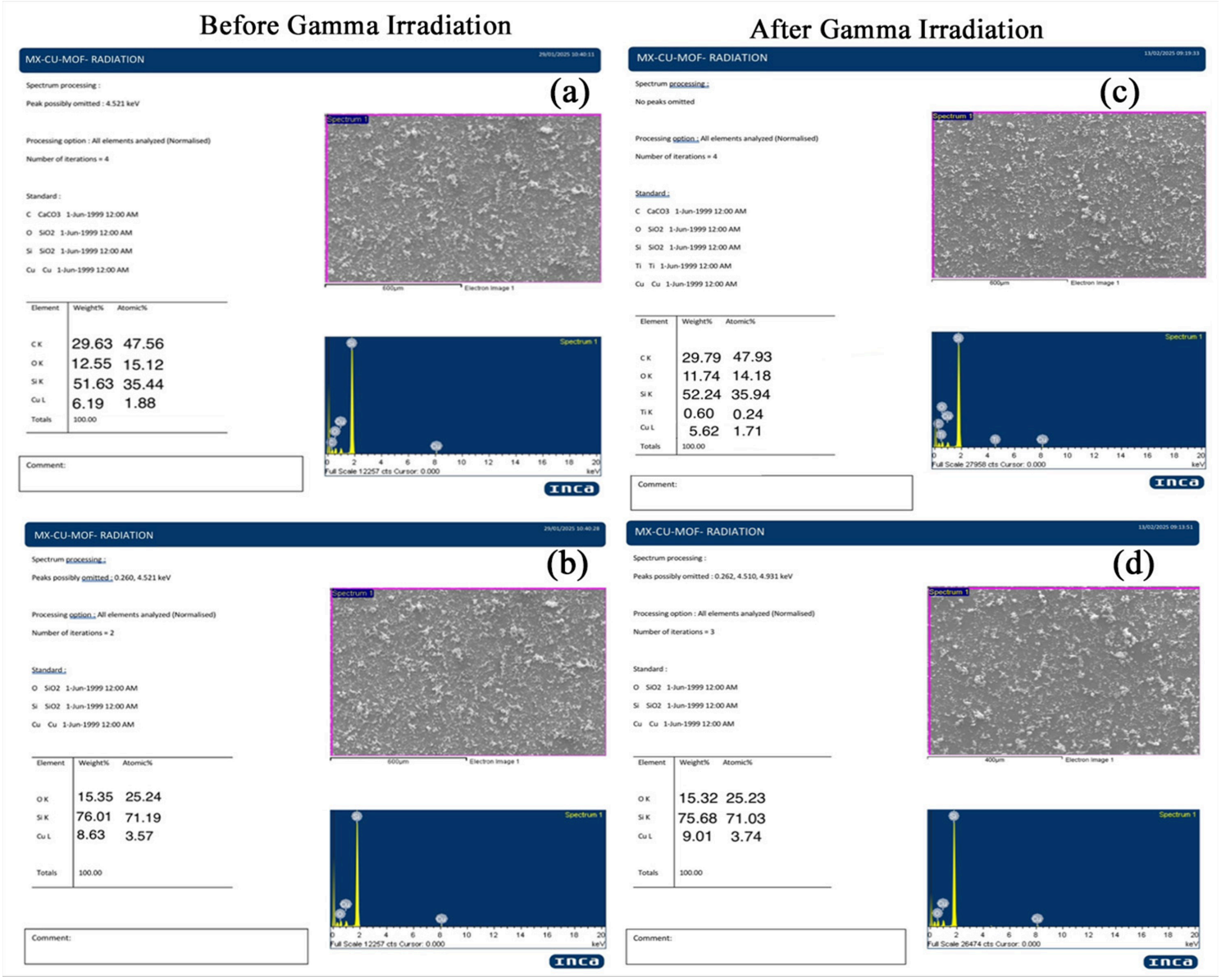


Figure 6

EDX spectra and corresponding semi-quantitative elemental compositions of representative regions of the MXene/Cu-MOF composite (a, b) before gamma irradiation and (c, d) after gamma irradiation.