

Synthesis and Antimicrobial Evaluation of Porous Al-Cu-Fe-Cr Quasicrystalline Powder via Ball Milling and Selective Leaching

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Abstract

In this research, the synthesis of porous Al-Cu-Fe-Cr quasicrystalline powder for antimicrobial properties was explored. An icosahedral formation Al-Cu-Fe base alloy was doped with 3 wt% chromium and then ball-milled for 50 hours at 250 rpm in the presence of n-hexane to inhibit oxidation. Then hot-pressed at 600 degree celsius done to form icosahedral phase. After this, a 10% NaOH solution was used to selectively leach the aluminum, inducing surface porosity. XRD confirmed the retention of icosahedral quasicrystalline phase. Antimicrobial activity testing against *Bacillus* species determined significant inhibited zones, showing potential biomedical significance.

This study demonstrates the formation and antimicrobial behavior porous quasicrystalline material through mechanical processing and chemical treatment.

Keywords: Al-Cu-Fe-Cr alloy, Ball milling, Porous structure, Antimicrobial activity, Quasicrystals

Introduction and Literature Review

Quasicrystals have drawn considerable attention due to their extraordinary structural, thermal, and functional features. Their non-stick, high-hardness and low-friction behavior has positioned them suitable for coatings and biomedical applications. However, their biological activity can be significantly improved through structural modifications such as elemental doping and surface porosity.

Recent work by Zahoor et al. (2020) demonstrated the antimicrobial potential of leached Al-Cu-Fe quasicrystalline powders, where alkaline NaOH leaching selectively removed Al, leaving behind Cu and Fe enriched surfaces with fine pores [4]. This enhanced surface activity was linked to improved antibacterial behavior against both Gram-positive (*Bacillus cereus*) and Gram-negative (*K. pneumoniae*, *E. aerogenes*) bacteria [4]. Importantly, the icosahedral structure was retained after leaching, confirming the structural stability of the QC phase.

In addition, mechanical alloying was carried out for up to 50 hours using high-energy ball milling. This duration was found to be necessary for producing refined beta phase nanostructure powder capable of forming stable quasicrystalline phases after hot pressing. This supports our choice of 50 hours of milling, especially since some studies have shown that 30 hours of milling results in only partial phase formation, which requires further structural confirmation using X-ray diffraction (XRD). [4].

While most prior studies focused on binary or ternary Al-based systems, newer research like Mohsin et al. (2025) expanded antimicrobial exploration into Ag-doped Al-Cu-Fe systems, confirming that selective leaching in NaOH enhances surface reactivity, and that elemental substitution (e.g., Ag, B, Co) boosts bacterial inhibition through synergistic effects of surface chemistry and microstructure. Their findings on $\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{11}\text{Ag}_1$ (structurally similar to our base composition) show that mild doping levels (0.5–2 at%) enhance antimicrobial action without compromising structural integrity [3].

Although Cr-doped Al-Cu-Fe quasicrystals are less commonly explored, our use of 3 wt% Cr aligns with substitution levels reported for other doping agents like B and

Ag, which were found effective for refining grain size, increasing Cu surface enrichment, and enhancing biological activity after leaching. Our sample composition is $\text{Al}_{65}\text{Cu}_{23}\text{Fe}_{11}\text{Cr}_3$ [3].

Methodology

Materials Preparation

A base Al-Cu-Fe powder was prepared with 3 wt% of chromium doping. The powders were mixed well before processing [2].

Quasicrystal formation

The powder mixture was subjected to milling in a planetary ball mill. Milling was for 50 hours at a rotational speed of 250 rpm. N-hexane was added periodically during milling to prevent oxidation. Successful milling without oxidation was further indicated by the absence of black coloration and the development of a uniform gray hue in the final powder [1]. Then the recovered powder is hot pressed at 600 degree Celsius and two pellets were annealed at 400 degree Celsius. Then hot pressed and annealed pellets were ground into fine powder to increase surface area [1].

Leaching Process

The powder was treated with a 10% NaOH solution. This leaching process selectively dissolved aluminum, creating a porous surface morphology. The powder was then thoroughly washed with distilled water and dried at room temperature [3]. While two powder samples were not leached and were only hot-pressed and annealed to compare the effectiveness among these powders.

X-Ray Diffraction (XRD) Analysis

XRD patterns for the ball-milled and leached powder were captured in order to verify phase stability. The diffraction peaks agreed with the reported icosahedral quasicrystalline structure, which confirms that the material's structural integrity was maintained through the process [3].

Antimicrobial Testing

The processed powder was evaluated for antimicrobial activity using agar diffusion tests. The sample was tested against *Bacillus* species. Powder samples were applied to the agar surface, incubated and the zones of inhibition were measured using a ruler [4].



Figure 1. Image of gray powder after milling

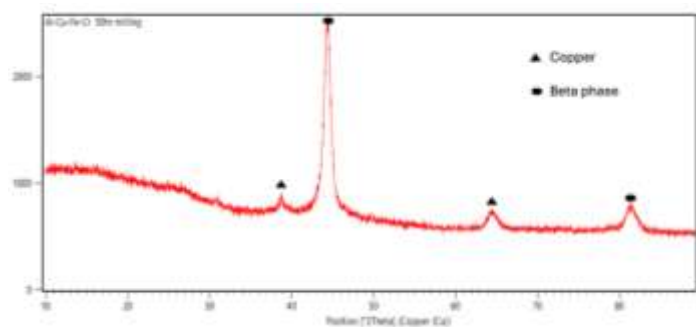


Figure 2. XRD pattern showing Beta phase



Figure 3. Zone of inhibition for Bacillus

Results and Discussion

Ball milling produced a gray, fine powder, which shows beta phase formation and no oxidation of powder because of the protective nature of n-hexane. The gray color transformation ensured that the milling process was effective. Selective leaching using 10% NaOH effectively removed aluminum from the QC matrix, resulting in increased surface area, which is expected to enhance antimicrobial performance. X-ray diffraction (XRD) analysis (Figure 2) confirmed the preservation of the icosahedral

quasicrystalline phase. The presence of copper peaks in the XRD pattern is attributed to incomplete diffusion of copper within the matrix during milling. This residual copper is anticipated to serve as a primary contact-killing agent upon subsequent leaching, during which it transforms into copper oxide which is an active component in antimicrobial mechanisms. The sharp diffraction peaks at characteristic angles further confirm that the long-range structural order of the alloy remains intact, even after extended milling and chemical processing. The antimicrobial activity test showed measurable zones of inhibition (Figure 3). The presence of clear halos around treated regions indicates strong antibacterial properties.

Table I				
Diameters of inhibition zones of bacillus bacteria				
Bac tria type	Hot press & leached (13)	Annea led & leache d (14)	Hot pres sed (12)	Annea led (11)
Bacillus	25mm	20mm	12mm	8mm

The hot-pressed and leached powder sample, labeled as number 13 in Figure 3, exhibited the largest inhibition zone. This is attributed to the leached surface, which exposes porous copper sites to the bacteria. The annealed and leached sample, labeled as number 14 in the same figure, showed a slightly smaller inhibition zone compared to sample 13. This reduction is likely due to the annealing process, which caused the copper present in the hot-pressed sample to become more diffused as a result of prolonged thermal exposure. The hot-pressed and annealed samples exhibited approximately equal diameters of inhibition zones (DIZ), both of which were smaller than those of the leached samples. This is due to the lower surface exposure of copper and a higher concentration of aluminum on the surface, which reduces porosity and active surface area [3]. The observed antimicrobial effect is attributed to the combined influence of increased surface area resulting from porosity and the incorporation of chromium, which enhances the material's surface reactivity. In the agar diffusion test, *Bacillus* species were used to evaluate the antibacterial performance of the quasicrystalline powder. Among the four differently processed powder samples, all exhibited notably larger inhibition zones compared to those reported in previous studies, such as Zahoor et al. (2020). These improved results highlight the effectiveness of chromium doping and surface structuring in disrupting bacterial cell walls. Furthermore, this approach offers a more cost-effective alternative to silver doping, which has been commonly employed in earlier studies. The comparatively larger inhibition zones observed in this study provide strong evidence of the enhanced antimicrobial activity [4].

Conclusion

A new porous Al-Cu-Fe-Cr quasicrystalline powder was successfully prepared. Chromium addition together with porosity on the surface greatly enhanced the antimicrobial

activity of the material. And the leached powder samples showed best inhibition zones. The icosahedral structure was retained as confirmed by XRD analysis. The methodology is easy, scalable, and offers a promising avenue to the development of bioactive materials for use in the environment and medicine.

Further research could consider a wider range of microbial. Future work will extend this study by evaluating the antimicrobial activity of the synthesized Cr-doped quasicrystalline powders against a broader spectrum of microorganisms, including both Gram-positive and Gram-negative bacterial strains as well as fungal species. This will allow for a comprehensive assessment of their bioactive potential across different microbial cell wall structures. By expanding the testing panel beyond *Bacillus* species, we aim to better understand the spectrum and mechanism of action of these porous materials, ultimately guiding their application in medical devices, coatings and environmental antimicrobial solutions.

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