# Etching-Time-Dependent Partial Conversion of $Nb_2AlC$ into $Nb_2C$-Containing Layered Materials Using NaF-HCl: Structural Evolution and Electrochemical Response

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## Abstract

The use of NaF-HCl as an etching medium offers an alternative route for transforming $Nb_2AlC$ into $Nb_2C$-containing layered materials. However, the influence of etching duration on structural transformation and electrochemical properties remains insufficiently understood. This study investigated $Nb_2AlC$ etching using NaF-HCl (6 M HCl) at 40 °C for 12, 24, and 90 h. XRD revealed a broad reflection at 2θ = 6.7° after 90 h, corresponding to an interlayer spacing of 1.32 nm. Residual $Nb_2AlC$ reflections indicated incomplete conversion. SEM showed progressive layer opening, while EDX confirmed a decrease in Al content from 12.06 at.% to 4.23 at.%. FTIR and Raman analyses further indicated changes in surface chemistry and bonding characteristics. The 90 h-modified electrode exhibited the highest anodic and cathodic current densities (3.743 and 3.392 mA $cm^{-2}$, respectively) and the lowest charge-transfer resistance (5416 Ω). SWV also demonstrated a linear glucose response over 0.1-0.4 mM ($R^2$ = 0.9954), providing preliminary evidence of its potential for electrochemical sensing.

Keyword: *$Nb_2C$, etching, electrochemical response*

## Introduction

MXenes are two-dimensional transition-metal carbides and nitrides, usually obtained by selectively removing the A-element layers from MAX-phase precursors. Their conductivity, layered structure, and tunable surface chemistry have drawn interest for energy storage, catalysis, and electrochemical sensing. Niobium carbide MXene, $Nb_2CT_x$, is of particular interest because its electronic structure and surface chemistry differ from those of titanium-based MXenes, and its surfaces show more uniform termination and greater resistance to oxidation.

Converting $Nb_2AlC$ into $Nb_2C$-containing layered materials requires removing Al while keeping the Nb-C framework intact. Direct HF etching is effective but raises safety and environmental concerns. Fluoride salts combined with HCl generate reactive fluoride species in situ and offer a safer alternative, as in the NaF-HCl system.

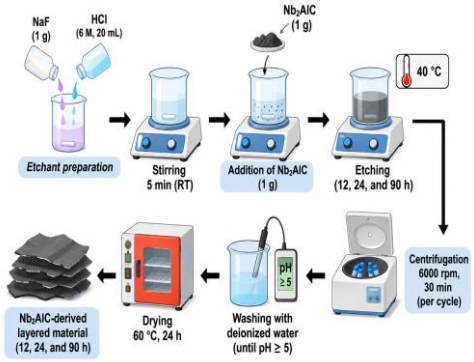
Etching duration strongly affects the extent of Al removal, interlayer expansion, and surface termination. Earlier studies on $Nb_2AlC$ used HF etching for 30 to 360 h and reported 168 h as the most effective, while other HF/HCl routes used 72 and 96 h. These results cannot be applied directly to NaF-HCl. How duration-dependent structural changes relate to electrochemical response in this system remains unclear.

This study examined NaF-HCl etching of $Nb_2AlC$ at 40 °C for 12, 24, and 90 h. Structural changes, charge transfer, and glucose response were assessed using XRD, SEM-EDX, FTIR, Raman, CV, EIS, and SWV.

## Research Method

### $Nb_2AlC$ etching process

NaF (1 g) was dissolved in 20 mL of 6 M HCl in a PTFE container under stirring for 5 min. $Nb_2AlC$ (1 g) was gradually added, and the mixture was maintained at 40 °C for 12, 24, and 90 h. The products were centrifuged and washed with deionized water until the supernatant reached pH ≥ 5, followed by drying at 60 °C for 24 h.

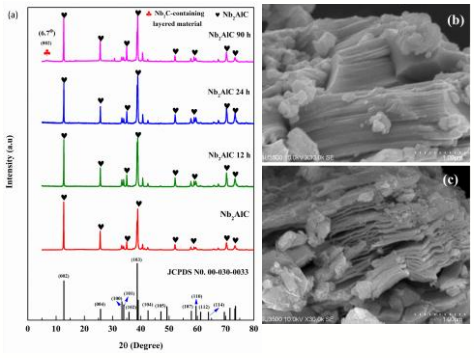



*Picture 1. Illustration of the partial convertion of $Nb_2AlC$ into $Nb_2C$-containing layered materials*

### Characterization and Electrochemical Measurements

Crystal structure was analyzed by XRD (Cu Kα). Morphology and elemental composition were examined using SEM-EDX, while surface functional groups were investigated by FTIR and Raman spectroscopy. Carbon paste electrodes (CPEs) were prepared from graphite and paraffin oil (7:3, w/w) and modified by drop-casting 10 µL of a 1 mg/mL aqueous material suspension.

Electrochemical measurements were performed using a three-electrode cell with Ag/AgCl and Pt wire as reference and counter electrodes, respectively. CV was recorded from -0.6 to +0.8 V in 0.01 M $[Fe(CN)_6]^{3-/4-}$ containing 0.1 M NaCl at scan rates of 5-50 mV/s. EIS was conducted at open-circuit potential over 0.1 Hz-100 kHz and fitted using a Randles equivalent circuit. SWV was performed using the 90 h-modified electrode in 0.1 M $KNO_3$ (0 to +0.7 V, 15 Hz). Statistical analysis used one-way ANOVA followed by Tukey's HSD test (α = 0.05).




*Picture 2. (a) XRD patterns of pristine $Nb_2AlC$ and etched samples; SEM images of (b) pristine $Nb_2AlC$ and (c) 90 h-etched material*

## Discussion & Result

XRD patterns of the 12 and 24 h samples remained dominated by $Nb_2AlC$-related reflections (**Picture 2a**). After 90 h, a broad (002) reflection appeared at 2θ = 6.7°, corresponding to an interlayer spacing of 1.32 nm, while the $Nb_2AlC$ reflection at 12.8° remained detectable. These features indicate partial conversion into an $Nb_2C$-containing layered material.

SEM revealed densely stacked layers in pristine $Nb_2AlC$ (**Picture 2b**), whereas the 90 h-etched sample exhibited more open, accordion-like structures (**Picture 2c**). EDX confirmed a decrease in Al content from 12.06 to 4.23 at.%, supporting progressive Al removal. FTIR and Raman further indicated changes in surface chemistry and bonding characteristics.

Electrochemical response improved with etching time. The 90 h electrode gave the highest anodic and cathodic currents and the lowest charge-transfer resistance (5416 Ω), compared with 9319 Ω for the bare electrode and 11433 Ω for $Nb_2AlC$. Only the 90 h electrode differed significantly from the bare electrode ($p < 0.001$), which suggests a threshold effect.

Furthermore, SWV showed an increasing anodic response with glucose concentration (0.1-0.4 mM), yielding a linear calibration with $R^2$ = 0.9954. These findings demonstrate that prolonged NaF-HCl etching promotes partial structural conversion and improves the electrochemical response of $Nb_2AlC$-derived layered materials

## Conclusion

Etching duration influenced the structural evolution of $Nb_2AlC$ during NaF-HCl treatment (6 M HCl, 40 °C). Samples etched for 12 and 24 h remained dominated by $Nb_2AlC$-related reflections. After 90 h, a broad reflection at 2θ = 6.7° indicated an expanded interlayer spacing of 1.32 nm, while the residual reflection at 12.8° confirmed incomplete conversion into $Nb_2C$-containing layered material.

SEM revealed more open, accordion-like layers after 90 h of etching. EDX showed a decrease in Al content from 12.06 to 4.23 at.%, while FTIR and Raman indicated changes in composition and bonding characteristics. These findings support progressive but incomplete Al removal.

The 90 h-modified electrode exhibited the highest anodic and cathodic current densities and the lowest charge-transfer resistance (5416 Ω). It was also the only electrode showing a statistically significant difference from bare CPE. Furthermore, it demonstrated a linear glucose response over 0.1-0.4 mM ($R^2$ = 0.9954).

Overall, prolonged NaF-HCl etching improved the interfacial electrochemical response of $Nb_2AlC$-derived layered materials without achieving complete Al removal. Further studies should focus on etching optimization, XPS analysis, selectivity, and validation using real samples.