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RSM-Driven Synthesis of Nickel Fluoroborate and Its Flame-Retardant Power on Cotton

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Abstract

Fluoroborates, a specialized class of boron compounds, possess diverse and significant applications, including their use as flame retardant additives, catalysts, and optical materials. This study focused on determining the optimal synthesis parameters for nickel fluoroborate and investigating its applicability to cotton fabrics. Nickel fluoroborate was synthesized using a wet chemical method with nickel oxide (NiO) and fluoroboric acid (HBF_4) as reactants, where the key parameters investigated were the HBF_4/NiO molar ratio, temperature, and reaction duration. The Response Surface Methodology (RSM) was employed for optimization, leading to the establishment of the following optimum synthesis conditions: a 4:1 molar ratio, a temperature of 90°C, and a duration of 100 minutes. Under these conditions, nickel fluoroborate was produced with a 98% yield and 89.5% purity, and the product was comprehensively characterized using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), Fluoroborate (BF_4^-) ion-selective electrode, X-ray Diffraction Analysis (XRD), and Fourier Transform Infrared Spectroscopy (FT-IR). In the subsequent phase, the product's application to cotton fabrics was assessed by treating samples with varying concentrations of the nickel fluoroborate solution for different immersion periods, with changes in the mass gain ratio being monitored. Crucially, the limiting oxygen index (LOI) test was performed, and the results conclusively demonstrated that the synthesized nickel fluoroborate exhibits excellent flame-retardant properties.

Key Words Nickel fluoroborate, Flame Retardant, Experimental Design, LOI

Introduction

Converting boron-based raw materials into high-value-added final products yields specialized boron compounds with a broad range of applications across various industries. These compounds are broadly categorized into three main groups: magnesium borates, sodium and calcium borates, and borosilicates (Yünlü, 2016). The mineral composition within boron deposits varies depending on the geological structure, with the most prevalent boron minerals being salts of sodium, magnesium, and calcium. While these minerals are chemically similar, their differing amounts of crystal water result in distinct properties (Özkan et al., 2016). Commonly utilized specialized boron compounds include boron nitride, zinc borate, boron phosphate, ferroboron, boron carbide, potassium borohydride, disodium octaborate tetrahydrate, sodium borohydride, boric acid, sodium tetraborate, and sodium metaborate (Yünlü, 2016).

Boron compounds derive their commercial value primarily from their tenor ratio, specifically the concentration of boron oxide (B_2O_3) present. While a small fraction of mined boron products is consumed directly as minerals, the vast majority is utilized to synthesize various downstream boron compounds (Yiğitbasıoğlu, 2004). The extensive utility of boron and its derivatives spans multiple high-technology sectors: they serve as a core raw material for glass fiber production (Kantürk, 2006) and in the manufacturing of heat-resistant glass (Markanda, 2023) they are critical components in the production of steel and composite materials (Ediz & Özdağ, 2001) they find applications in the defense industry (Bilgiç et al., 2024), nuclear power plants (Özkan et al., 2016), and as ingredients in aircraft and rocket fuels (Meydan, 2019) furthermore, they are essential for hydrogen production and storage (Yenmez, 2011), function as effective flame retardant additives (Shen, 2014), are employed in the treatment of cuts and burns (Khaliq, 2025), and are broadly utilized across the agricultural sector (Çetinkaya et al., 2014; Çopur et al., 2023), among many other diverse fields.

Fluoroborates, recognized as high-value-added specialty boron products, are compounds with diverse applications as flame retardants, optical materials, and catalysts. Numerous methods have been developed for synthesizing fluoroborate compounds. Generally, fluoroborates are obtained by reacting boric or fluoroboric acid with metal halides, oxides, carbonates, bicarbonates, or fluorides. An alternative method involves reacting boron fluoride with metal fluorides to yield stable fluoroborates (Y. Aydın et al., 2021). These compounds boast broad

utility as flame retardant additives, catalysts, pesticides against insects and fungi, and as chemical materials that enhance the optical properties of glass (Olgun et al., 2023). Specifically, ammonium fluoroborate is widely used in industrial applications, including as a flame retardant chemical additive, in metal processing and soldering, for separating water and methanol from ethanol in the food sector, and in pest control (Ceyhan et al., 2020). Zinc fluoroborate has extensive applications in the textile industry, chemical synthesis, plating baths, and glass technologies. It is frequently used as an additive, particularly in flame-retardant coating compositions for synthetic fibers. It is also favored as a catalyst in chemical reactions due to its high yield, short reaction time, and high selectivity. Furthermore, potassium fluoroborate minimizes smoke generation, enhancing flame resistance in halogenated (chlorine and bromine-containing) and halogen-free polymers (Y. Aydın et al., 2017).

Fluoroborates are prominent specialty compounds distinguished by their inherent flame retardancy and smoke suppression capabilities. The recent rise in fire incidents has amplified the global demand for advanced flame-retardant materials, underscoring the critical need for scientific and technological research to develop products that offer superior protection, enhanced long-term durability, and robust environmental compatibility. Functionally, flame-retardant materials act as a protective barrier that inhibits ignition, slows the propagation of flames, and mitigates smoke generation (Manivannan, 2025). When incorporated as additives, these materials work by diluting the combustibility of the host substrate and increasing its limiting oxygen index (LOI) (Fanfarová et al., 2016). Boron-based flame retardants create a glassy protective layer that shields the polymer chains from oxidation. Crucially, boron-containing compounds are considered environmentally friendly in flame-retardant applications because they do not contribute to the emission of toxic gases during combustion (Gürü et al., 2021). To accurately assess the fire resistance of treated materials, various standardized tests are conducted, the most common of which include the limiting oxygen index (LOI) test, the flame spread test, the smoke density test, and flammability tests (Durgun, 2010).

This study aims to determine the synthesis parameters of nickel fluoroborate, a compound classified as a specialty boron product, and to investigate its flame-retardant properties on cotton fabrics. To the best of the authors' knowledge, no prior research has been published regarding the determination of the synthesis parameters or the evaluation of the flame retardant characteristics of nickel fluoroborate.

Materials and Method

The synthesis of nickel fluoroborate was executed using the wet chemical method. Various quantities of nickel oxide (NiO 97% purity, sourced from Acros Organics) and fluoroboric acid (HBF_4, 50% purity, sourced from Acros Organics) were utilized as reactants. Nickel fluoroborate synthesis reaction is given in Eq. 1.

$$NiO + 2HBF_4 \longrightarrow Ni(BF_4)_2 + H_2O \quad (1)$$

A reflux apparatus was designed to mitigate the environmental impact of fluoroboric acid volatilization during the exothermic reaction and to facilitate its recovery into the system. The experimental parameters investigated were the reactant molar ratio ($nHBF_4/nNiO$) at levels of 4:1, 8:1, and 12:1; temperature at 30°C, 60°C, and 90°C; and reaction time at 20, 60, and 100 minutes. The stirring speed was maintained constant at 350 rpm (rotations per minute) throughout the experiments. Experimental design was conducted using the RSM, and 19 sequential experiments suggested by the method were performed, based on the varying parameters. The maximum theoretical yield of nickel fluoroborate was calculated according to the reaction stoichiometry in Equation 1, and the actual yield was determined from the amount of product synthesized. The synthesized nickel fluoroborate was characterized using ICP-OES (Agilent 5800), BF_4^- ion-selective electrode (Mettler Toledo DX287), XRD (GNR APD 2000 Pro), and FT-IR (Shimadzu IRSpirit-X) analyses. For the application of nickel fluoroborate to cotton fabrics, the resulting mass gain on the fabrics was investigated at various concentrations and dipping times. The LOI test, performed according to the ASTM D2863-00 standard, evaluated the flame-retardant property. Nickel fluoroborate synthesized under optimum conditions was prepared at different concentrations (200 g/L, 300 g/L, 400 g/L), and the fabric samples were impregnated with the solutions and subsequently dried. The properties of the cotton fabric used in these experiments are presented in Table 1.

Table 1. Properties of the fabric used in the LOI test

Fiber type	**%100 cotton**
Density of field	520 g/m^2
Weaving type	2x2
Rope size	15 pieces/cm in the weft direction
	23 pieces/cm in warp direction

Results and discussions

The initial phase of the experimental investigation focused on determining the impact of various parameters on the reaction yield. Experiments were conducted according to different sets suggested by the RSM, utilizing reactant conditions set at molar ratios ($nHBF_4/nNiO$) of 4:1, 8:1, and 12:1; temperatures of 30°C, 60°C, and 90°C; and reaction times of 20, 60, and 100 minutes. The resulting products, initially in solution form, were subsequently filtered and dried. It was observed that when the reaction temperatures were low, specifically 30°C and 60°C, unreacted nickel oxide was present in the product phase, and the formation of crystals was minimal.



Fig. 1 The obtained magnesium fluoroborate crystals.

ICP-OES analysis

ICP-OES analysis was performed to quantify the nickel and boron concentrations in the synthesized products. To calculate the reaction yield, the concentrations of boron and nickel in the initial solution were determined using the measured ICP-OES concentrations in conjunction with the dilution factors of the products. Subsequently, the amount of nickel fluoroborate was calculated. The calculated yield based on the nickel fluoroborate quantity derived from the analysis results showed excellent concordance with the experimentally obtained solid yield of the synthesized product.

Under reaction conditions at 30°C, the failure of nickel oxide to react completely resulted in the formation of a precipitate, consequently leading to a significantly low yield. Experiments conducted at 60°C showed a relative increase in yield, with a further, more substantial increase observed at 90°C. Specifically, the reaction yield under the optimum conditions suggested by the RSM ($nHBF_4/nNiO$=4:1; 90 °C; 100 minutes) was calculated to be 98.07%.

Table 2. The reaction conditions and yield suggested by the RSM

STD	Run	Temperature (°C)	Mol ratio ($nHBF_4$/nNiO)	Time (min)	Rxn efficiency (%)
1	1	30	4	20	0,13
3	2	30	4	100	0,18
16	3	60	8	60	6,14
10	4	90	8	60	61,13
5	5	30	12	20	0,12
19	6	60	8	60	7,93
9	7	30	8	60	0,20
4	8	90	4	100	98,07
13	9	60	4	60	5,64
18	10	60	8	60	4,92
7	11	30	12	100	0,21
2	12	90	4	20	68,52
11	13	60	8	20	1,98
15	14	60	8	60	6,60
12	15	60	8	100	8,97
14	16	60	12	60	5,66
6	17	90	12	20	78,59
17	18	60	8	60	6,37
8	19	90	12	100	77,63

Purity analysis

The purity analysis of the nickel fluoroborate synthesized via the wet chemical method was conducted using a BF_4^- ion-selective electrode. For comparison, a reference solution was prepared by dissolving a specified amount of the commercial standard, Acros Organics nickel fluoroborate (98% purity), in 250 ml of water. This reference solution's BF_4^- ion concentration was measured by an ion meter, and the data were recorded. Subsequently, an identical mass of the synthesized nickel fluoroborate crystals, produced under optimum conditions, was dissolved in 250 ml of water, and its ion concentration was measured using the same BF_4^- ion-selective electrode. To enhance the accuracy of the measurements, 1 ml of ionic strength adjuster (ISA) solution was added for every 50 ml of the prepared solutions. The measured ion meter values for the synthesized and commercial nickel fluoroborate solutions (prepared using the same masses and volumes) were compared. The analysis results determined that the synthesized sample exhibited a purity of 89.5%.

Optimization studies with RSM

Optimization studies were carried out using the RSM for experimental design, leveraging the demo version of the Design-Expert 11 software. This methodology defined temperature, molar ratio ($nHBF_4/nNiO$), and time as the variable parameters, with yield as the response variable. The model's compatibility was confirmed by the data obtained from the program, as the difference between the Predicted R^2 value (0.7951) and the Adjusted R^2 value (0.9676) was less than 0.2, indicating good agreement. Furthermore, an Adequate Precision value greater than 4 signifies that the model is suitable for navigating the design space. According to the RSM results, the highest yield was achieved under the following optimal synthesis conditions: a temperature of 90°C, a reactant molar ratio ($nHBF_4/nNiO$) of 4:1, and a duration of 100 minutes. The predicted yield from the synthesis under these optimum conditions, as determined by the model, is 91.423%.

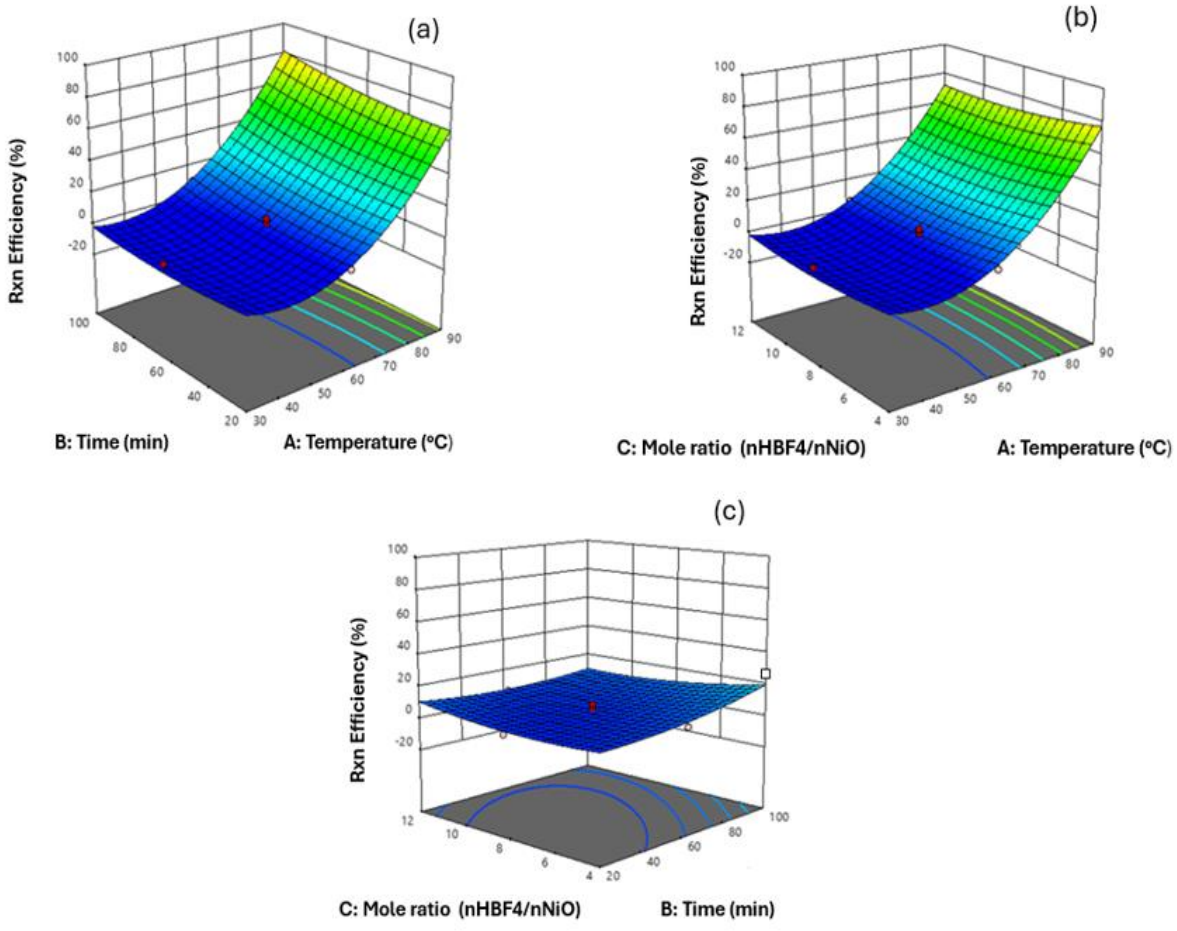


Fig. 2 (a) 3D representation of the effect of temperature and time on yield,

(b) 3D representation of the effect of temperature and molar ratio on yield,

(c) 3D representation of the effect of molar ratio and time on yield.

The 3D response surface plot shown in Fig. 2(a), which illustrates the interaction between temperature and time on the reaction yield, indicates that increasing the temperature consistently led to improved yield. Furthermore, the plot suggests that the region where the reaction time was set to 60 minutes yielded better results than other time settings, according to the methodology. Examining the effect of the temperature and molar ratio interaction on yield in Fig. 2(b), temperature is clearly identified as the parameter with the most significant influence on reaction efficiency. In contrast, the molar ratio exhibits a more limited effect. Finally, the plot in Figure 2(c), which depicts the interaction between time and molar ratio, shows that this combination does not significantly impact the final yield.

FT-IR analysis

FT-IR analysis was performed to identify the functional groups of the synthesized and commercially obtained nickel fluoroborate. The results of the FT-IR analysis are presented in Fig. 3.

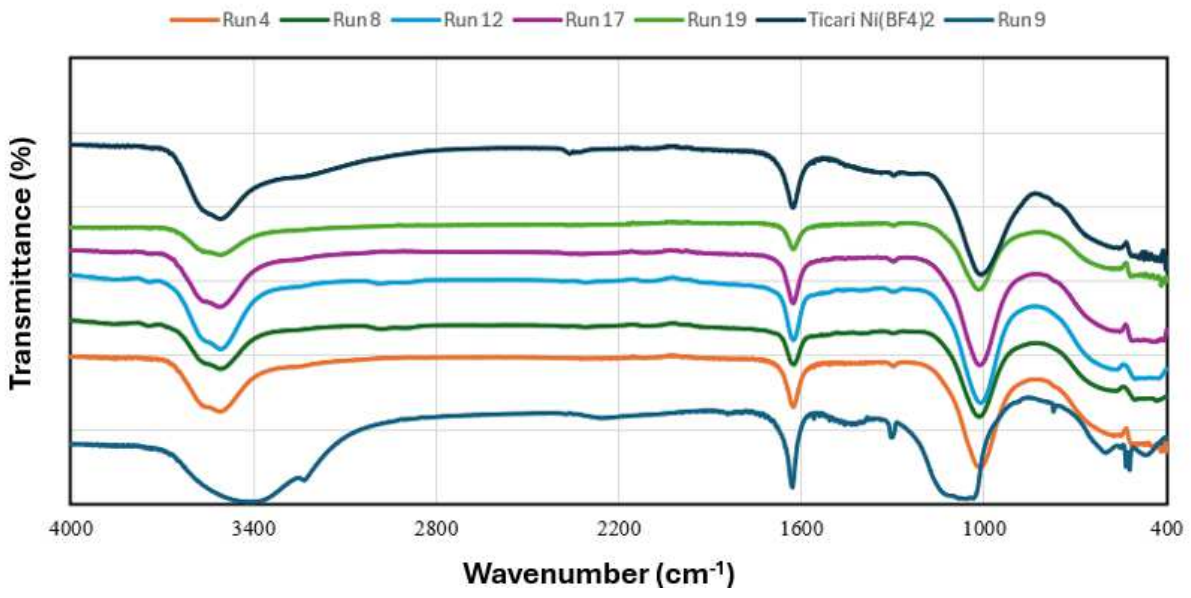


Fig. 3 FT-IR spectra of nickel fluoroborate synthesized under different experimental conditions

Literature indicates that the characteristic B-F bond vibration for fluoroborate compounds is typically observed in the 1000-1100 cm^{-1} range. An analysis of the FT-IR spectra revealed a high degree of concordance between the characteristic peaks of the synthesized product and

those of the commercial standard, thereby confirming the successful production of nickel fluoroborate. Minor discrepancies and the presence of new peaks are understood to indicate the presence of impurities. Specifically, the characteristic B-F bond was clearly observable in the 1000-1100 cm^{-1} range across the spectra. Concurrently, vibrational peaks corresponding to the O-H bond were detected in the 3200-3500 cm^{-1} region (Dai et al., 2023; Leoni et al., 1991; Cintrón & Hinchliffe, 2015).

XRD analysis

In this study, XRD analysis was performed to obtain information about the molecular structure of the synthesized nickel fluoroborate and the commercially obtained nickel fluoroborate.

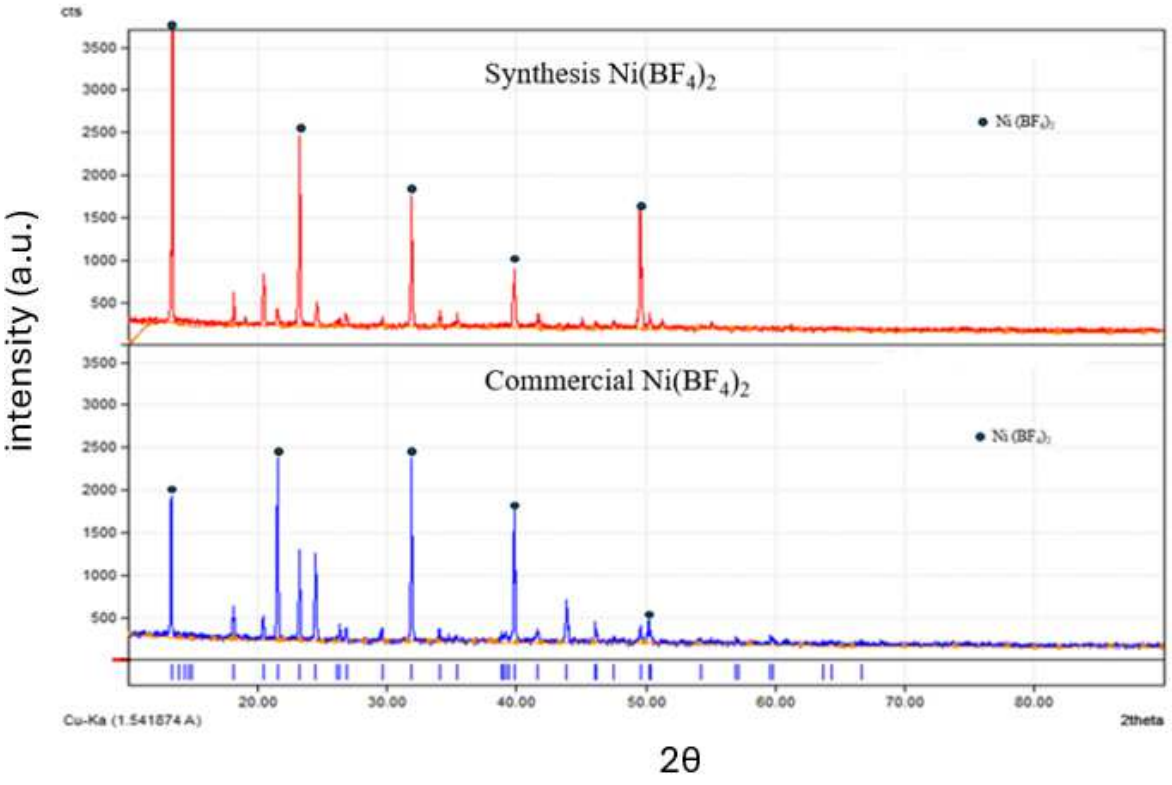


Fig. 4 Comparative XRD pattern of commercial nickel fluoroborate and nickel fluoroborate synthesized under optimal conditions

According to the graph in Fig. 4, the XRD peak values for the nickel fluoroborate synthesized under optimum conditions demonstrate a high degree of agreement with those of the commercial nickel fluoroborate. However, the difference in impurities between the two

products negatively impacts the intensity of some peaks. Furthermore, the synthesized nickel fluoroborate was observed to possess a crystal lattice structure, with its average crystallite size (D) measured as 90.38 nm, while the crystallite size (D) of the commercial nickel fluoroborate was determined to be 89.44 nm. A comparative XRD graph illustrating the results for the synthesized nickel fluoroborate and the nickel oxide used as a reactant, as per Eq. 1, is provided in Fig.5.

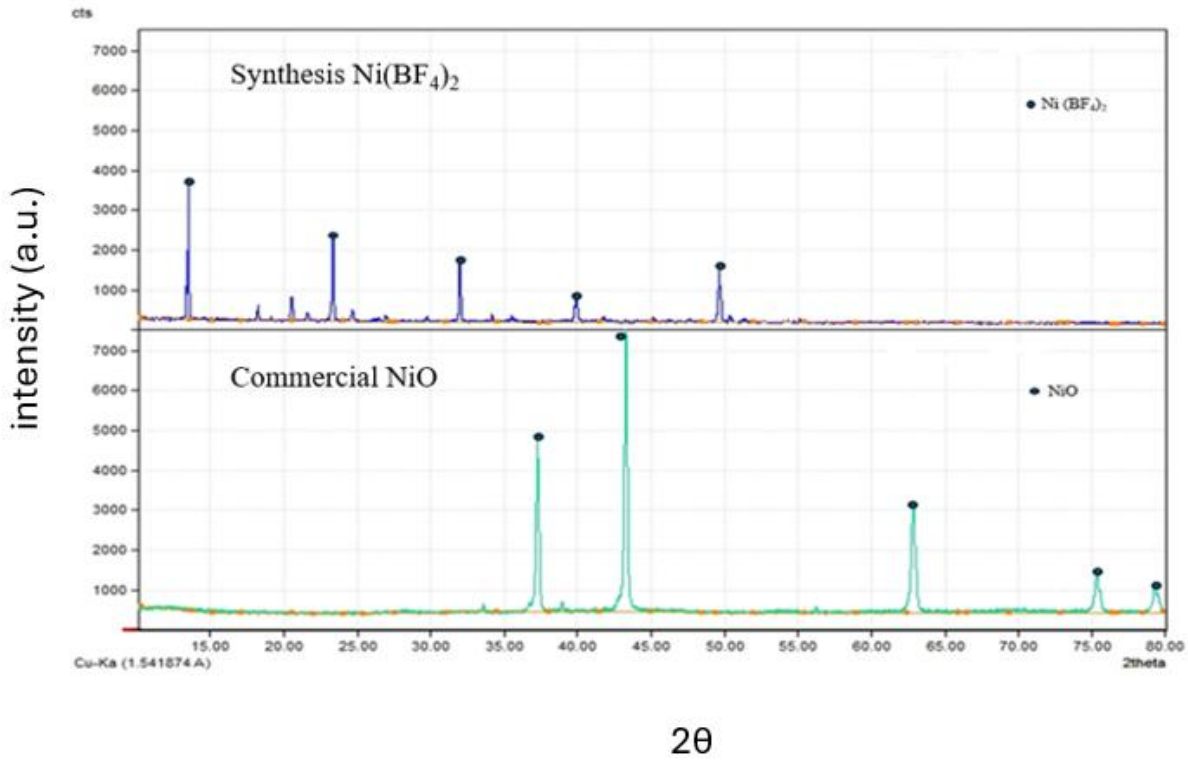


Fig. 5 Comparative XRD pattern of commercial nickel oxide and nickel fluoroborate synthesized under optimal conditions

The analysis results indicate a difference between the peak values of the synthesized product and the nickel oxide used as the reactant. Crucially, the absence of nickel oxide peaks in the final nickel fluoroborate product obtained from the experiment confirms that the reaction successfully reached completion.

Applicability Studies on Cotton Fabrics and LOI Test

The final stage of the study involved investigating the applicability of the nickel fluoroborate synthesized under optimum conditions using the wet method onto cotton fabrics. Nickel fluoroborate solutions prepared at various concentrations (200 g/L, 300 g/L, 400 g/L) were impregnated onto high-basis-weight cotton fabric samples for varying immersion times (15 minutes, 30 minutes, 60 minutes). The resulting changes in the mass gain ratio were

analyzed. At a fixed immersion time of 15 minutes, increasing the concentration from 200 g/L to 300 g/L resulted in a mass gain increase from 12.6% to 20.55%, and further expanding it from 300 g/L to 400 g/L raised the mass gain ratio from 20.55% to 29.7%. Under conditions where the immersion time was held constant at 30 minutes and 60 minutes, the mass gain ratio on the fabrics increased with rising concentration, confirming the successful incorporation of nickel fluoroborate into the fabric structure. Consequently, concentration was identified as a critical parameter affecting the percentage mass gain on cotton fabrics and for the synthesized product prepared at a 200 g/L concentration, 15, 30, and 60 minutes of immersion resulted in mass gains of 12.6%, 12.75%, and 12.8%, respectively. No significant increase in mass increase rates was observed when the fabrics were immersed in NiBF4 solutions at 300 g/L and 400 g/L concentrations for different periods. This result indicates that the time factor has a limited effect in these experiments and suggests that processing at varying durations may not be necessary.

LOI tests were conducted based on the ASTM D2863-00 standard to determine the flame-retardant properties of the nickel fluoroborate on cotton fabrics. The required 100% cotton fabric samples were prepared in 10x2 cm dimensions and impregnated with nickel fluoroborate solutions at 200 g/L, 300 g/L, and 400 g/L. In the tests performed according to the ASTM D2863-00 standard, a flame of constant size was applied to the top edge of the vertically positioned specimen for 30 seconds. Subsequently, the flame was withdrawn every 5 seconds to observe whether the specimen would continue to burn independently (self-sustain the combustion). It is noted that an LOI value above 28% is generally required for a material to be considered flame-retardant (Durgun, 2010). The data obtained from the LOI test results are presented in Table 3.

Table 3 Mass gain percentages and LOI values of cotton fabrics treated with nickel fluoroborate at varying concentrations

Sample name	Mass gain (%)	LOI value (%)
Untreated fabric	-	15
Fabric impregnated with 200 g/L $Ni(BF_4)_2$ solution	12.8	30
Fabric impregnated with 300 g/L $Ni(BF_4)_2$ solution	21.7	33
Fabric impregnated with 400 g/L $Ni(BF_4)_2$ solution	31	35

The results presented in Table 3 indicate that the LOI value for the untreated cotton fabric was 15%. Since this value is lower than the oxygen content in air (21%), it demonstrates that the pristine material possesses an easy-burning characteristic. Conversely, the LOI values for the cotton fabrics treated with varying concentrations were measured as 30%, 33%, and 35%, respectively. Based on these data, as the solution concentration increased, the mass gain ratio and the fabrics' LOI value rose, demonstrating an enhancement in the fabrics' flame retardancy property. It was therefore concluded that high basis weight cotton fabrics, such as tent materials impregnated with nickel fluoroborate, acquire flame retardancy even in environments with a high oxygen index.

Conclusions

In this study, nickel oxide and fluoroboric acid were used as reactants for synthesizing nickel fluoroborate, and the synthesis was carried out using the wet method. Optimization studies were performed based on the RSM to determine experimental parameters, focusing on the effects of reactant molar ratio ($nHBF_4/nNiO$), temperature, and reaction time on the yield. Optimal production was achieved with a 98% yield and 89.5% purity under conditions where the reactant molar ratio ($nHBF_4/nNiO$) was 4:1, the temperature was 90°C, and the reaction time was 100 minutes. XRD and FT-IR analysis confirmed the successful synthesis, showing a high degree of agreement in peak values between the synthesized product and commercial nickel fluoroborate. In the application phase of the study, the nickel fluoroborate produced under optimum conditions was impregnated onto cotton fabrics. An increase in solution concentration led to a significant mass gain on the fabrics, while the immersion time was found to have a negligible effect on this mass gain. Subsequent LOI tests demonstrated the remarkable flame-retardant properties. The LOI value of the untreated fabric was 15%, whereas the samples treated with 200 g/L, 300 g/L, and 400 g/L concentrations yielded LOI values of 30%, 33%, and 35%, respectively. These results confirm that nickel fluoroborate is an effective flame retardant material, suggesting its widespread use in the textile and related industries.

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Declarations

Conflict of interest. The authors declare no conflict of interest.

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