Novel Ti3C2-Nickel Phosphate Hybrids: Synthesis, Characterisation, and Antimicrobial Mechanisms

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**Abstract:** MA-TiC-Nickel-Phosphate hybrids have been reported as novel antimicrobial materials which can be obtained through a chemical vapor deposition of Ti₃C₂ MXene and nickel phosphate by hydrothermal method. These hybrids are described using techniques such as TEM, SEM, XRD and spectroscopy and further established to cause cell membrane rupture, oxidation and the release of ions that can invariably kill bacteria. This new material can be used in coating and other related applications especially in medical fields and in the purification of water. Antibacterial performance is boosted with the help of photothermal and photodynamic treatments, indicating good perspectives for the fight against MDRA and for increasing the rates of people’s wellbeing and protection throughout the whole civilized world.The goal of this study is to develop and to investigate Ti₃C₂-Nickel Phosphate composites, to determine their antibacterial effects and to discuss possible applications in coatings and films, medical equipment and drinking water treatment.In order to obtain an MXene phase of Ti₃C₂, a 20 ml of hydrofluoric solution is mixed with Ti₃AlC₂ and placed on a shaker for 24 hours at 40C. The mixture is than, through centrifugation, brought to pH 7 proceeding to wash it with ethanol and lastly, dried at a temperature of 80C for 24 hours to obtain black Ti₃C₂. Separately, 2. 326 g of nickel nitrate and 2.The reactions between the reagents takes place in stages, and the total quantity of reagents is calculated by the resulting excess of the reagent which gives less amount of product. The required quantity of approximate 2713 g of the disodium hydrogen phosphate is dissolved in water with constant stirring for half an hour. Phosphate solution is mixed into nickel nitrate solution resulting to a formation of a light green precipitate, the solution is then stirred for full one hour. Ti₃C₂ is then added into water and finally added into the precipitate solution and stirred for 3 hours. The received mixture suspension is subjected to microwave treatments and centrifuged, washed and subsequently dried at 80°C for 24 h; and finally calcined at 300°C for 3 h, which results in black Ti₃C₂ nickel phosphate.The reviewed study related the synthesis of Ti₃C₂ MXene and Ti₃C₂ nickel phosphate composites, which were noted for their high antibacterial effects linked to the cell membrane rupture and oxidation. Characterization validated their fabrication, and the materials exhibited applications in generating coatings, medical applications entities and water purification services with special incentive on multidrug-resistant bacteria and hence enhancing health standards.The Application of FTIR of Ti₃C₂-Nickel Phosphate hybrid displayed critical antimicrobial chemical bonds and groups such as Ti-C, Ti-O, P-O,-OH, and Ni-O indicating the hybrid’s structural and functional effectiveness. Nevertheless, because of sophisticated issues related to synthesis and stability of the hybrids, these hybrids can be expected to perform a role of antimicrobial agents. The upcoming studies should focus on understanding the antimicrobial properties, growing concerns, action routes, and outcomes on the environment for better coverages.

**keywords:** Phosphate hybrids, MX- bacteria interaction, dimensional functional nanomaterials, tetramethylammonium hydroxide

# INTRODUCTION

TiC-Nickel Phosphate hybrids' synthesis and characterisation provide new directions for the creation of cutting-edge antibacterial materials. Optimizing the design and use of these hybrids in biological sectors will be made easier with an understanding of the antibacterial mechanisms.The flexible and varied composition of MXene, a recently developed multidimensional two-dimensional (2D) material, comes from surface-modified carbide. With a typical formula of Mn+1XnTx, where n = 1-3, they are composed of layers of early transition metals (M) interspersed with n layers of carbon or nitrogen (denoted as X), and ended with surface functional groups (denoted as Tx/Tz). (1) [(Zamhuri et al., 2021)](https://paperpile.com/c/q12zdi/JnPi5) MXenes resist biofouling TiClTx, which makes them good candidates for water desalination membranes. A study found that Ti₃C₂Tx killed more E. Coli and B. Subtilis bacteria than graphene oxide (GO). Ti₃C₂Tx killed almost 98% of bacteria in four hours at 200 μg/mL. This antibacterial effect happens because it damages the cell membrane, as LDH TEM, and SEM tests showed. TiCl3-Tx works well for water treatment because it kills bacteria and doesn't get clogged up by biological growth. (2) [(Rasool et al., 2016)](https://paperpile.com/c/q12zdi/X8zQO)There are some recent TiC-nickel phosphate hybrids in which scientists have been interested in as to how they are synthesized, what components they are built with, and how they can eliminate bacteria.[(Aparna et al., 2021; Poornima et al., 2021; Verma & Muthuswamy Pandian, 2021)](https://paperpile.com/c/q12zdi/cAXzD+WSxDD+lVmc4) To synthesise these hybrids, they incorporate Ti3C2 MXene with nickel phosphate through chemical vapour deposition and hydrothermal process(Rafi et al., 2024). To investigate the hybrids’ structure and shape they have employed tools like TEM, SEM, XRD and spectroscopy.[(Aparna et al., 2021; Poornima et al., 2021; Verma & Muthuswamy Pandian, 2021)](https://paperpile.com/c/q12zdi/cAXzD+WSxDD+lVmc4) These hybrids are good at killing bacteria, and this happens in a few ways: they disrupt cell membranes, induce oxidation and create the release of metal ions.Infectious diseases due to pathogens are a notable and increasing threat to global health.[(Merchant et al., 2022; Pandiyan et al., 2022)](https://paperpile.com/c/q12zdi/Yjp5f+e19vI) Furthermore, negligence towards prescribed antibiotics has escalated the emergence of some resistance microorganisms in a given community or a health facility.[(Aparna et al., 2021; Poornima et al., 2021; Verma & Muthuswamy Pandian, 2021)](https://paperpile.com/c/q12zdi/cAXzD+WSxDD+lVmc4) In this work, we employ intercalation and delamination method under the assistance of tetramethylammonium hydroxide (TMAOH) to obtain two-dimensional Ti3C2 nanosheets which have good biocompatibility and efficient photothermal-photodynamic synergistic antibacterial performance to E. coli and S. aureus. The combined photo-thermal/photodynamic antibacterial efficacy of Ti3C2 nanosheets is also significantly enhanced than that of the normal Ti3C2 nanosheets obtained by simple exfoliation using HF or situ HF (HCl+LiF). (3) [(Wang, J., Xuan, J., Liu, Y., Li, Z., Han, Y., & Wang, Z., 2023)](https://paperpile.com/c/q12zdi/dUC54) MXenes, an upcoming class of two dimensional functional nanomaterials, have as of recent past interest as potential nanomedicines (i. e., as antibacterial materials) because of the rapidly increasing instances of Multi-Drug Resistant (MDR) microbes (Tuluwengjiang et al., 2024). Recently, the membrane disruption effect and a high light-to-heat conversion efficiency of Ti3C2 MXene have been reported; nevertheless, due to MX- bacteria interaction, low photothermal therapy efficiency and secondary bacterial infection in vivo, its antibacterial property is still unsatisfactory.(4) [(Zheng et al., 2022)](https://paperpile.com/c/q12zdi/kINRO)New TiC-nickel phosphate composites have great potential in solving several challenges in material chemistry and AMR, thus, more investigation is needed. The requirement for new and efficient antimicrobial remedies is born from the novel infections that are not easily treatable due to high levels of resistance against several drugs. When nickel phosphate is mixed with TiCl3, MXenes which are already known for their different mechanical, chemical and electrical properties, a material with enhanced antibacterial properties can be obtained. It, therefore, implies that the improvement of the structure and functionalities of these hybrids can only be achieved with the knowledge of how they are synthesized and characterized. On the same note, if the exact antimicrobial mechanisms were clearly understood, then better materials might be developed for the above contexts that include coatings, medical devices and water purification hence enhancing health and safety among the population. The first sign of the Ti3C2Tx MXene nanosheets is antibacterial photothermal treatment effectiveness that decreases with size. Using ultrasonication, three MXene suspensions were created: There is a small one called MX-s, medium one called MX-m, and the large one is called MX-l. Ma was the small size was 196 nm. As mentioned the change in temperature of MXene suspensions (10 μg/mL) after 5 minutes of NIR irradiation was to 64 , 60 , and 56 °C for MX-s, MX-m, and MX-l, respectively. Thus, the correspondingly calculated viability loss of MRSA due to being irradiated by MX-s, MX-m, and MX-l under NIR was 93%, 69%, and 56%, respectively. (5) [(Gao et al., 2022)](https://paperpile.com/c/q12zdi/ZwrXC)MRSA is among the antibiotic resistant bacteria that exist and are dangerous to human beings. Great effort and focus should be devoted to the creation of treatments like PTT as well as PDT that do not involve the use of antibiotics. To achieve the most optimal PTT and PDT therapy, in this study, ICG is incorporated into Ti₃C₂Tx MXene nanosheets with a size of 454 nm. Neither plain MXene, ICG, or ICG-MXene did not show any detectable bacterial inhibition towards MRSA without NIR stimulation. In NIR, relative to the initial state, MRSA’s viability was reduced by 66% in ICG, 100% in ICG-MXene, and 45% in MXene. PTT and PDT mechanisms of MXene and ICG are held responsible for this enhanced effect and it is evident that MXene can effectively treat bacterial infection.. (6) [(Chenhao Yu, Shangyan Sui, Xiaotong Yu, Wenlong Huang, Yafei Wu, Xin Zeng, Qiaming Chen, Jun Wang, 2022)](https://paperpile.com/c/q12zdi/5Q1vc)

# MATERIALS AND METHODS

## Synthesis of Ti3C2 MXene

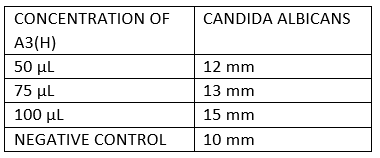
HF(20ml) is added to Ti3AlC2 and has ggto be stirred using a magnetic stirrer for 24 hours at 40 degree celsius. The Al will get evenly removed and the washing process is done in centrifuge to remove any contamination until Ph7 is obtained and then washed with ethanol twice. The precipitate obtained is then dried in the hot air oven for 24 hrs at 80 degree celsius and Ti3C2 is obtained which is black in color.[(Muthuswamy Pandian et al., 2022)](https://paperpile.com/c/q12zdi/h6eL)

2.326 gm nickel nitrate is added to 50ml distilled water (Solution A), Disodium hydrogen phosphate is used to obtain phosphate (2.2713gm)(Solution B) both separately stirred for 30 mins. Buret containing Disodium hydrogen phosphate is added to nickel nitrate solution drop by drop and precipitate is formed i.e, light green (Solution C) and stirred for 1 hr to get homologous solution. 1 gm Ti3C2 is added to 25 ml of distilled water and stirred for 20 mins dispersed (Solution D). [(Solanki et al., 2023)](https://paperpile.com/c/q12zdi/hzxC)Add this solution in solution C drop by drop and stir for 3hrs. Using the microwave method, the obtained solution D is placed in the microwave and is done for 10 mins by giving 2 mins interval in between.[(Muthuswamy Pandian et al., 2022; Ramakrishnan et al., 2023)](https://paperpile.com/c/q12zdi/h6eL+63AgZ) The precipitate is then taken in the centrifuge and the washing process is done to remove contamination using water 3 times, ethanol is used twice and acetone twice.[(Jain & Verma, 2022; Marya et al., 2022)](https://paperpile.com/c/q12zdi/Nev8W+VewBP)The precipitate obtained is dried in hot air oven at 80 degree celsius for 24 hrs, calcination is done at 300 degree celsius for 3 hrs then black powder Ti3C2 nickel phosphate is obtained.



1. (b)

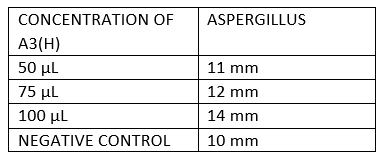
**Figure 1:** (a) (b) Synthesis of nickel phosphate & titanium carbide- nickel phosphate

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1. (b)

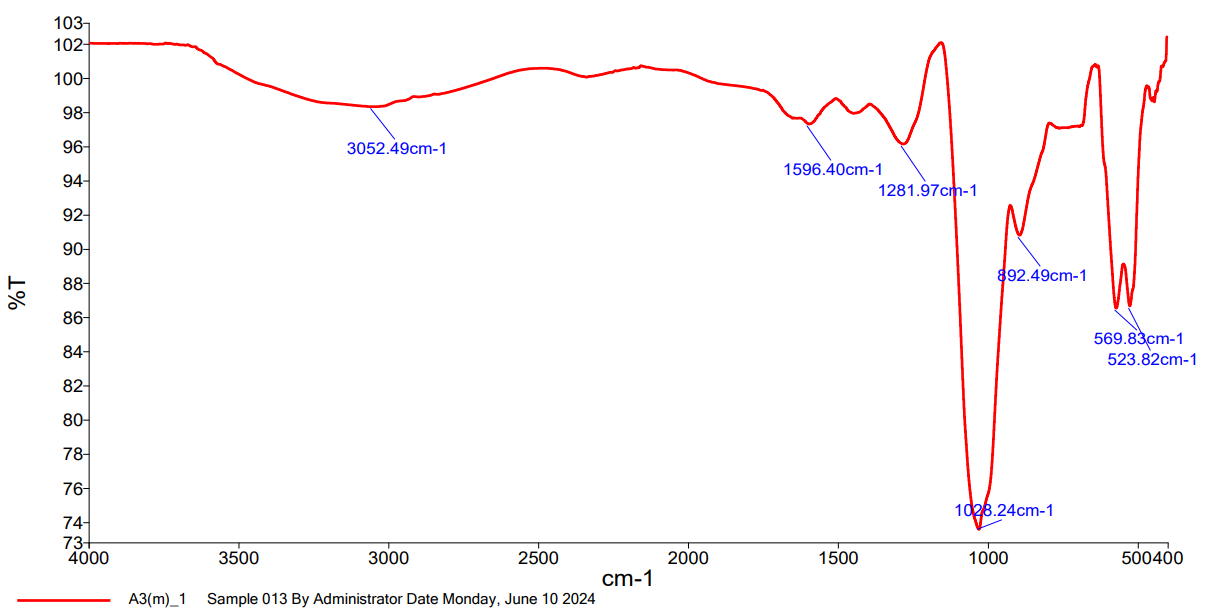
**Figure 2:** (a) (b) Antifungal Activity Of Ti3c2-Nickel Phosphate/ A3(H) / Candida Albicans

# RESULTS AND DISCUSSIONS

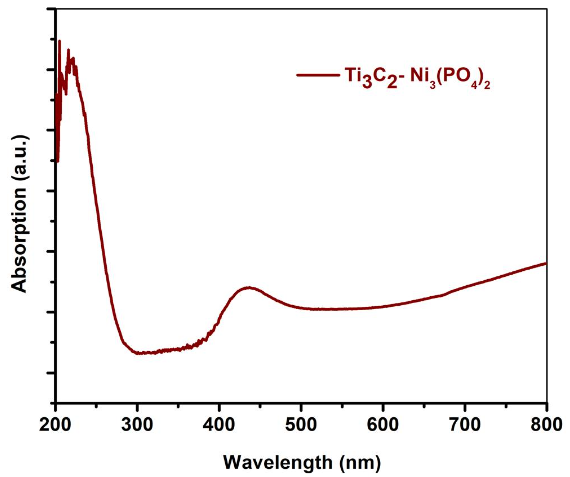


1. (b)

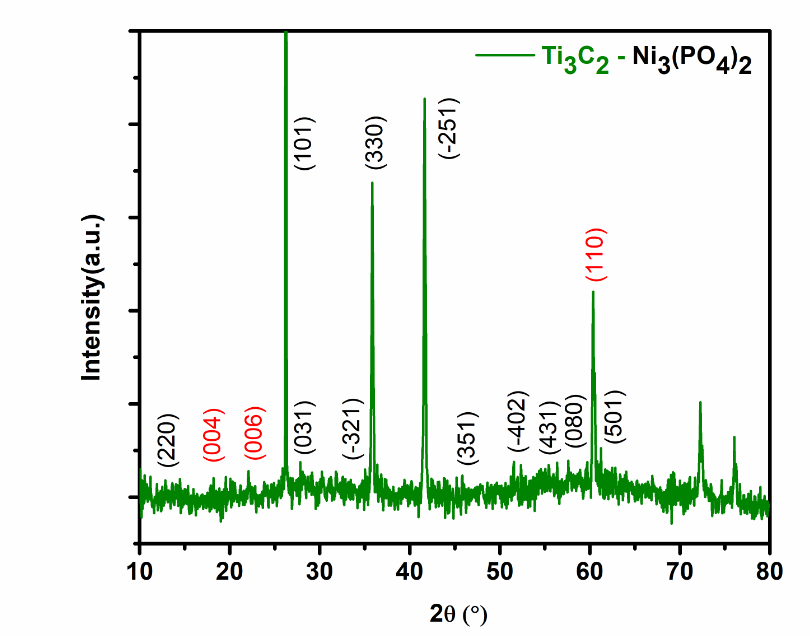
**Figure 3:** (a) (b) FTIR spectrum of Ti₃C₂/Nickel Phosphate Composite



**Figure 4:** UV vis DRS



**Figure 5:** UV vis DRS



**Figure 5:** XRD Pattern of Novel Ti₃C₂ -Nickel Phosphate Hybrids

The FTIR analysis of the novel Ti3C2-nickel phosphate hybrid proves to be shared of several characteristic peaks wherein the structure of the hybrid is shed light upon. [(Sreevarun et al., 2023)](https://paperpile.com/c/q12zdi/8v92M).The peaks at about 500-800 cm⁻¹ and 600-700 cm⁻¹ are typical for Ti-C and Ti-O bonds which points to the presence of the Ti₃C₂ MXene.[(Ganapathy 2022)](https://paperpile.com/c/q12zdi/Yjp5f+e19vI+Q59N1)[(Chokkattu et al., 2022; Ramamurthy et al., 2022)](https://paperpile.com/c/q12zdi/58H0t+7VqwE)On the same note, P-O stretching features of nickel phosphate can also be identified by the peaks at around 900-1100 cm⁻¹. The broad peak around 3200- 3600 cm⁻¹ indicates the presence of hydroxyl groups which could be because of the functionalization of the surface or the presence of adsorbed water. A few peaks in the range of 400-600 cm⁻¹ might be attributed to the Ni-O bonding that shows the interaction of the nickel ions with the MXene surface. Altogether, these peaks substantiate the existence of Ti₃C₂ MXene and nickel phosphate in the composite and the surface chemical groups that provide chemically stability and reactivity to the composite. This study conducted in 2020 by Szuplewska et al. highlights the use of MXenes, its derivatives and composites in bioimaging, nanomedicine as well as in the environmental field. Optical and electrochemical properties as well as stability of MXenes have been enhanced in the recent past. (7) [(Szuplewska et al., 2020)](https://paperpile.com/c/q12zdi/nU3vO)

Another aspect regarded to the structure of the material can be discussed based on the results of the X-ray diffraction (XRD) pattern of the synthesized Ti₃C₂-nickel phosphate hybrid. The feature (002) peak in the range of 6-9° (2θ) corresponds to the layered structure of the obtained MXenes. And it is found that individual diffraction peaks are attributed to nickel phosphate phases based on the crystalline structure. Intercalation effects can lead to peak shift, for instance shift of the (002) peak of Ti₃C₂ to the left which represents the increased interlayer spacing. Narrow and well-defined peaks indicate the presence of large crystallites while broad and less defined peaks indicate either the presence of small crystallites or non-crystalline phases. Variation in peak intensities provides additional information on the ratio and the reaction of the two hybrids, and thereby, successful formation of the composite, and interaction between the two hybrids.

The effectiveness of the synthesized Ti₃C₂-nickel phosphate hybrids towards Candida albicans is checked using normal microbiological methods. Possible ways of action are: The alteration of cell membranes, producing Reactive Oxygen Species (ROS) and changing the processes of metabolism. UV-vis DRS analysis gives information on the optical characteristics of the hybrids; band gap energy values, cooperative interactions induced by hybrid formation and probable photocatalytic ability of the hybrids. They also pointed out that better light absorption and interfacial charge transfer processes are beneficial for the material’s performance, indicating that it could be suitable for use in different applications.

As in our study we have a transitional metal (Nickel) and also a non-metal (Phosphate as it consist of both phosphorus and oxygen both being non metals). A study in 2019 discusses the ways to modify MXenes by doping MXenes with transition and non-metals, single-atom immobilized MXene, and the combined method of doping and immobilizing are effective in enhancing the catalytic performance of MXenes compared with the intrinsic MXenes. The data presented in the review showed that the doped MXenes possess higher activity, selectivity, stability, and improved properties compared with the pristine MXene in the applications pointed out in this work.[(Adel et al., 2023)](https://paperpile.com/c/q12zdi/zbhvV) At times, they also proved to be significantly more effective than commercial catalysts, currently in the market. (8) [(Tianyu Zhan a , Haoyong Yin a, \* , Jiajie Zhu a , Junli Chen a , Jianying Gong a , Ling Wang b , Qiulin Nie a, 2019)](https://paperpile.com/c/q12zdi/7iDlU)

In this study a microwave assisted method was used to synthesize 3D flower like hydration nickel phosphate (N-90).[(Wadhwani et al., 2022)](https://paperpile.com/c/q12zdi/2Fz3C) With the highest specific capacitance of 162 F/g, the N-90 electrode showed also the best cycling stability. In an asymmetric supercapattery with controlled rGO, it obtained an energy density of 25. 48 Wh/kg and power density of up to 750. Hence, its rating stands at 02 W/kg, making it desirable for use in supercapattery devices. (9) [(“Ni3(PO4)2-Coated Li[Ni0.8Co0.15Al0.05]O2 Lithium Battery Electrode with Improved Cycling Performance at 55 °C,” 2011)](https://paperpile.com/c/q12zdi/Ntsl8) The use of non-parenthetic Ti3C2Tx MXene during the preparation synthesis of high-quality carbide-derived GQDs utilized a solvothermal process. They came to realized that the nature of the DMF solvent medium was that it could act as a nitrogen doping agent as well as a solvent and that the microscopic structure of the GQDs could be very shiny and luminous nitrogen-doped GQDs. (10) [(L. Zhou, F. Wu, J. Yu, Q. Deng, F. Zhang, G. Wang, 2017)](https://paperpile.com/c/q12zdi/j1kGr)

In a recent study in 2020 we have discovered unbroken TiC fibers that don't stick together. They did this by using electrospinning on a mixture of tetrabutyl titanate, acetylacetone pure water, and polyvinylpyrrolidone (PVP). This way of making TiC fibers can also help to make other transition metal carbides, both as powders and fibers. (11) [(Lei Yua,∗ , Wangjin Jia,b , Shiwen Zhanga , Yun Songa , Hui Liua,b , Zhefei Wanga , Quan Liua , Xuhong Wang, 2020)](https://paperpile.com/c/q12zdi/7y6Ai)

# CONCLUSION

Analyzing the FTIR spectra of Ti₃C₂-Nickel Phosphate hybrids, some of the most important chemical bonds and functional groups associated with antimicrobial characteristics are identified. Teding of peaks for Ti-C, Ti-O, P-O, -OH, and Ni-O confirms the structural and functional efficiency of the developed composite. However, there are challenges that; synthesis of this material proves to be complex, and the material’s stability is a challenge for further research. More future work should be directed toward the explanation of the antimicrobial behaviors, the establishment of larger-scaled applications, increasing pathways of the antimicrobial action, and providing evaluations on the environmental effects. All in all, these results imply a promising application of Ti₃C₂-Nickel Phosphate hybrids as antimicrobial agents as long as these materials are further studied and fine-tuned for a wide variety of purposes.

# LIMITATIONS AND FUTURE SCOPE

While TiC-Nickel Phosphate hybrids have shown promising antibacterial activity, it is important to keep in mind the disadvantages of this material, namely the complicated synthesis of the hybrid substance and need for precise control over the reaction conditions as a consequence of which is prone to failure in large scale production. Moreover the durability of the hybrid compound in the long term is also problematic due to its fluctuating environmental conditions. Though the structural integrity of the hybrid compound can be confirmed by the FTIR and XRD spectroscopy experiments, further studies are needed to understand exactly the degradation mechanism and shelf life of this compound.

Furthermore, more studies are necessary to better understand the antimicrobial activities of the hybrid, including interactions with microbial cells and the exact role of reactive oxygen species formation. Also the study was limited to in vitro microbiological techniques; a study in vivo to confirm biocompatibility and usefulness of the hybrid should be done. The aim of the future study should include increasing material stability, simplifying the synthesis method and improving the study into environmental and biological uses.

Further investigations should be directed towards delineation of the molecular working of the antimicrobial property of the Ti₃C₂-Nickel phosphate hybrids, chronic toxicity test, investigation on performance of the material against more diverse microbial isolates, environmental influence test, and synergistic antimicrobial interaction studies for the purpose of confirmation of rejection of microbial resistance.

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