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Green synthesis and characterization of nickel oxide nanoparticles using *Psidium guajava* leaf

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Abstract
Green synthesis of NiO NPs with biological systems is becoming a growing field, especially in plant extracts nanotechnology. Biological reducing agents have been interpreted worldwide to lessen the impact of toxic chemicals applied in development of nanoparticles. In present research, work deals with green synthesis & characterization of NiO NPs via *Psidium guajava* leaf extract. SEM analysis confirmed that the highly agglomerated spherically shape of the synthesized nickel oxide nanoparticles. The TEM images indicated that Ni NPs had a homogeneously spherical shape and found to support the size reduction and confirmed that the synthesized nanoparticles are in nanometer range. EDX results confirmed the presence of nickel and oxygen in the synthesized NiO NPs. FT-IR studies clearly showed the formation of NiO and indicated that the plant extract contains various phytochemicals. Also, the UV was performed to investigate the optical nature of the prepared NiO nanoparticles.

Keywords: *Psidium guajava* leaf extract, Nickel oxide nanoparticles, Green synthesis, FTIR; Transmission Electron Microscope

1. Introduction
Psidium guajava, popularly known as guava, is a small tree of the myrtle family. It is employed in medicinal ingredients traditionally to advance the exterior wound healing process. Also, guava is proven to be a medicinal plant to have abundant biological functions, like: anti-cough, anti-diabetic, anti-bacterial, antispasmodic, and anti-oxidant properties. The leaves have several compounds that perform as fungi static & bacteriostatic agents, and it also possess significant quantity of antioxidants & owns radio protective capability. The guava leaves extracts are supplemented with photochemical biological composite such as vitamins, flavonoids, and phenolic compounds, catechia, gallic acid, which act as reducing & stabilizing agents during the preparation of nanoparticles. Guava leaves minimize breast, prostate, & oral cancers risk, because of high amounts of antioxidant, Lycopene. Numerous studies shown that lycopene acts a substantial role in reducing cancer risk. It contains huge number of organics & inorganics such as tributary metabolites, e.g., polyphenols, antioxidants, antivirals & anti-inflammatory composites. The several parts of plant are employed in general medicine to control malaria, vomiting, diarrhea, dysentery, ulcers, etc. Biological/living resources/ landscape, like: Plants & micro-organisms, can be employed in a facile, safe, quick & cost-effective. The green method related to medicinal plants is fetching a good line to the synthesis of nanoparticles. The plant extract is used in the green path as capping and reducing agents. Herein, an effort is being made to prepare NiO NPs using *Psidium guajava* leaf extract was carried out.

Nanotechnology is one of the most interesting and challenging research fields in biomedical engineering, modern materials science, water treatment and pharmacology due to its unique and attractive physicochemical properties. Metal nanoparticles have many applications, such as antioxidant potency, antimicrobial activity against human pathogens, use in food products, anti-inflammatory activity in cosmetics, cancer diagnostics and therapy, and DNA detection and cell labeling. Mu *et al.* (2011) [4] found that adding three metal oxide nanoparticles (nano-TiO_2, nano-Al_2O_3, and nano-SiO_2) to Anaerobic Digestion did not inhibit CH_4 generation, while nano-ZnO was inhibitory at doses > 6 mg/g TSS. Unsar and Perendeci (2018) [7] investigated Al_2O_3 nanoparticles for anaerobic digestion of waste sludge and were found that there is 14.8% increase in CH4 at 250 mg/g TS of Al_2O_3 NPs. The trace metal elements such as Co, Cu, Fe, Mo, and Ni could play significant roles in stimulation as well as stabilization of AD of waste organic materials when they are present at low concentrations (Abdelsalam *et al.*, 2016; Luna-del Risco *et al.*, 2011) [1, 3].

Currently, plant extracts can be used as reducing reagents to rapidly synthesize nickel nanoparticles. Plant extracts are not only a good way to make benign and stable nanoparticles but also a good way to decrease the use of toxic chemicals or strong Reductant. However, the synthesis of stable and good-sized metal nanoparticles using different plant extracts is somewhat challenging. To the best of our knowledge, this is the first report to synthesize nickel oxide nanoparticles using *P. guajava* L. leaf extracts at room temperature. In the current work, we reported a simple and rapid method for the synthesis of nickel nanoparticles by a one-step reduction of nickel ions using *P. guajava* L. leaf extracts. The physicochemical characteristics of the synthesized nickel oxide nanoparticles were analyzed using various techniques (UV-vis, FTIR, SEM with EDX, TEM, and zeta potential analysis).

2. Materials and Methods

2.1 Materials: *Psidium guajava* (*Guava*) leaves were collected from Courtallam, Tenkasi District Tamil Nadu for the extraction of leave extract for the formation of NiO nanoparticles. Nickel chloride dehydrate of 99% purity, Whatman No.1 filter paper and distilled water are used for this research work. The working solutions were prepared by using distilled water and also the glassware were thoroughly washed with it. All chemicals that utilized in this research work are of analytical grade with no further purification.

2.2 Preparation of *Psidium guajava* leaf extract: *Psidium guajava* leaves were collected from our college premises and thoroughly cleaned with running tap water to remove debris and other contaminants, followed by distilled water and then shade dried to take away the remaining moisture at room temperature. The aqueous extract of the leaves was prepared by taking 100 g of leaves and were cut into small pieces then grind in pestle and mortar by adding little amount of measured 250 ml of distilled water and kept in water bath at 60°C for 4 hours until the colour of the aqueous solution changes to brown red. After being cooled, the leave extract was filtered with Whatman No.1 filter paper and filtrate was stored at 4°C in a refrigerator in order to be used for further experiments.

2.3 Green synthesis of NiO Nanoparticles

To synthesize NiO nanoparticles, the precursor solution was prepared by adding 8g of nickel chloride in 100 ml of distilled water and this was added drop wise to the filtrate of 100 ml kept under magnetic stirrer for continuous mixing of the solution until the colour changes. After cooling down to room temperature, the resulting solution was incubated overnight. Then obtained solution was centrifuged at 3500 rpm for thrice by washing with distilled water. The resulting precipitate was collected and dried in hot air oven at 105°C till the moisture was removed. The finally obtained nickel oxide nanoparticles were carefully placed in a clean air tight sample holder for further characterization of the NiO nanoparticles. The green synthesized NiO nanoparticles from Guava leaves shown in Fig 1.

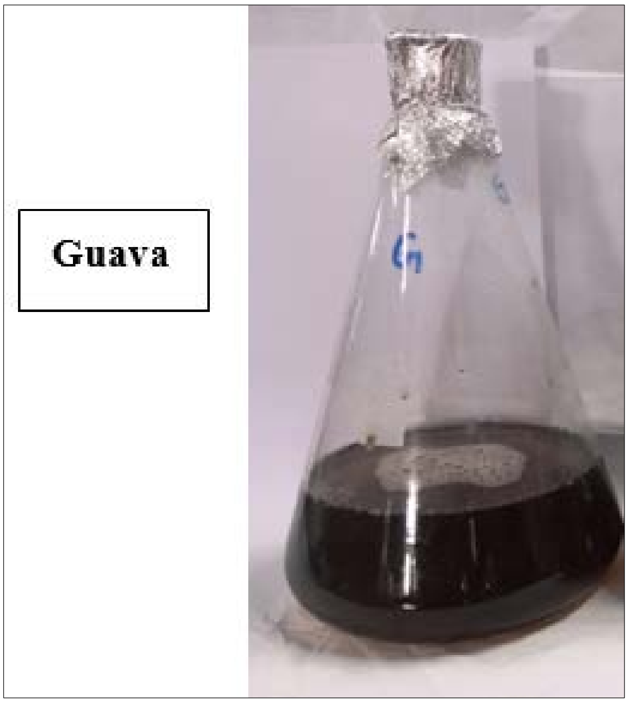

Fig 1: Green synthesized NiO nanoparticle from guava leaves

2.4 Characterization of NiO nanoparticles: Synthesized NiO nanoparticles capped with *Psidium guajava* leaf extract were inspected by FTIR, SEM, TEM, EDAX, UV, and particle size analyzer. The identification of functional groups of selected leaf extract and NiO-NPs were confirmed using FTIR spectroscopy (IR Prestige-21 Shimadzu). FTIR Spectroscopy (FT/IR-6800 Attenuated Total Reflect ant Unit ATR pro one sensor) is an analytic method to recognize the polymeric, organic, and inorganic materials. The possible functional groups are examined using Fourier Transform Infrared Spectroscopy (Shimadzu, Japan) infrared (IR) double beam spectrophotometer. The SEM (JEOL JSM 6360 LA, Japan) was performed to visualize the size & shape of nanoparticles. The synthesized NiO NPs was placed on a carbon-coated copper grid and observed under SEM EDX and TEM microscopes. The absorption spectra of NiO-NP were measured using Shimadzu 2600 UV-Visible spectrophotometer. The particle size of NiO-NPs was measured by particle size analyzer (PSA) Malvent Zeta sizer. To estimate the particle size distribution and zeta potential, a colloidal nickel nanoparticle solution was measured using a Zeta sizer Nano Instrument (Malvern, Britain) at 30°C with a 90° detection angle.

3. Results and Discussion

The peak value at 330.00 nm specifies the absorption of metal ions in guava leaves using UV-Vis shown in Fig. 2. Ezhilarasi *et al.* (2018) [2] obtained the similar results for green synthesis of NiO nanoparticles using *Aegle marmelos* leaf extract. Wardani *et al.* (2019) [8] and Uddin (2021) [6] obtained the synthesis of NiO nanoparticles via green route using *Ageratum conyzoides* L. leaf extract and berberi stem. Similar results were obtained from Sabouri *et al.* (2021) [5] for green synthesis of nickel oxide nanoparticles using *Salvia hispanica* L. (chia) seeds extract. Guava leaf extract shows FT-IR spectra at the wave number 1739 cm^{-1} as C-N stretching indicating that leaf extract contains alkaloid as shown in Fig. 3. The particle size of NiO nano particle obtained from guava leaves was found to be 296 nm given in Fig. 4. Scanning electron microscopy (SEM) analysis confirmed that the highly agglomerated spherically shape of the synthesized nickel oxide nanoparticles were found and shown in Fig. 5 and EDAX image shows an existence of Ni and O elements throughout the composition was confirmed by the achieved result shown in Fig. 6. The morphology and distribution of nanoparticles were characterized using TEM (Transmission Electron Microscope). The TEM images indicated that Ni NPs had a homogeneously spherical shape and found to support the size reduction and confirmed that the synthesized nanoparticles are in nanometer range shown in Fig. 7. The EDAX image of guava leaves shown in Fig. 8.

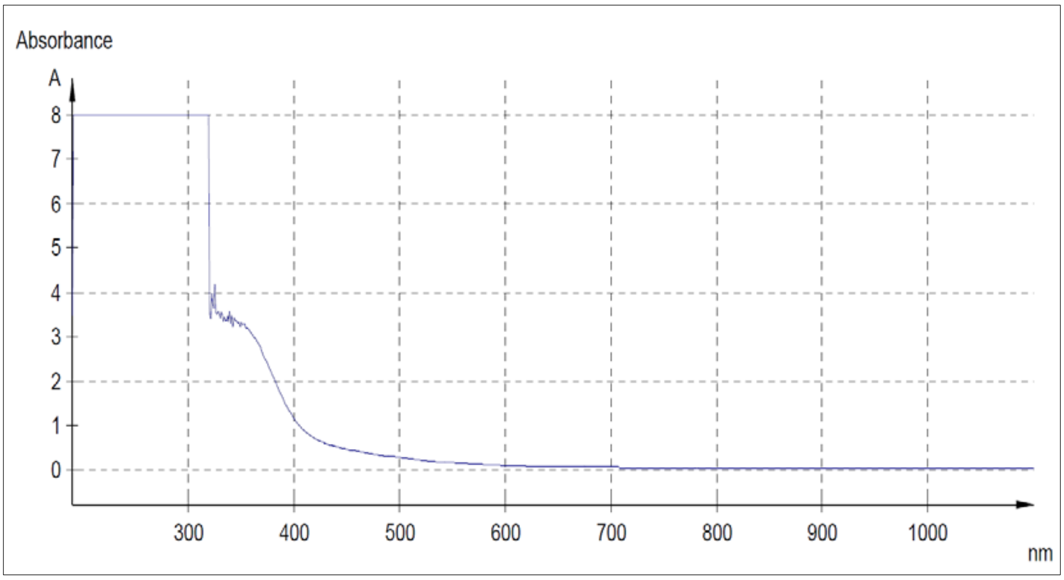

Fig 2: UV-Vis of guava leaves

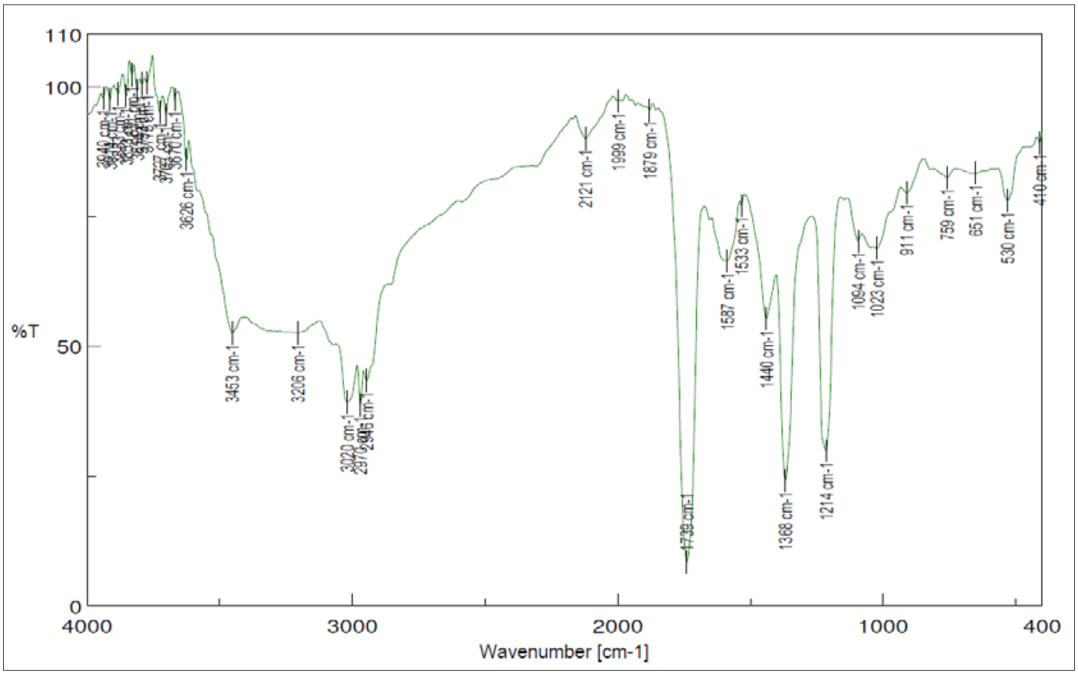

Fig 3: FTIR of guava leaves

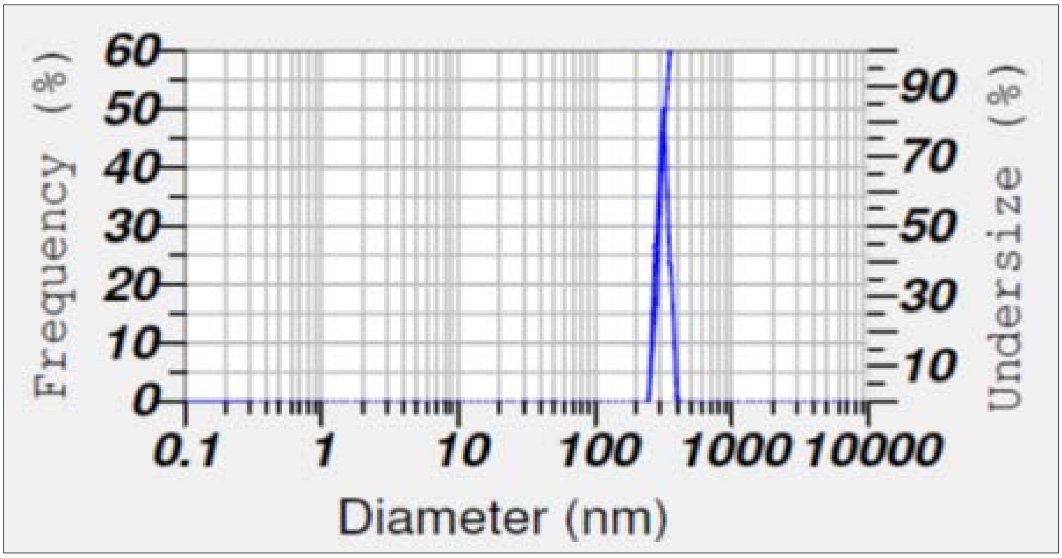

Fig 4: Particle size for guava leaves

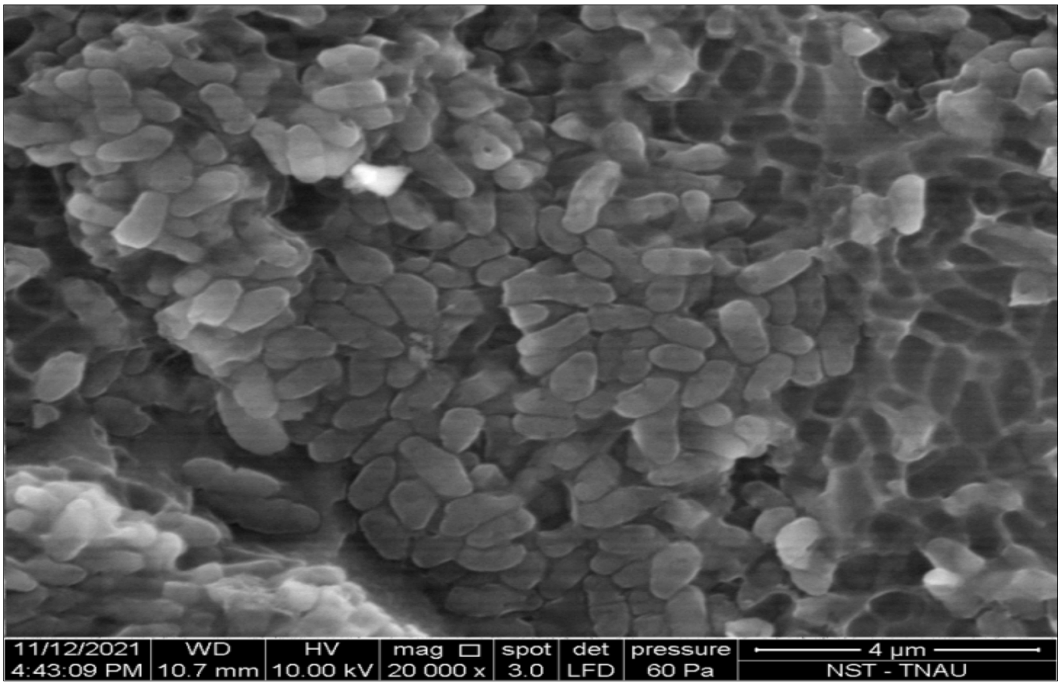

Fig 5: SEM for guava leaves

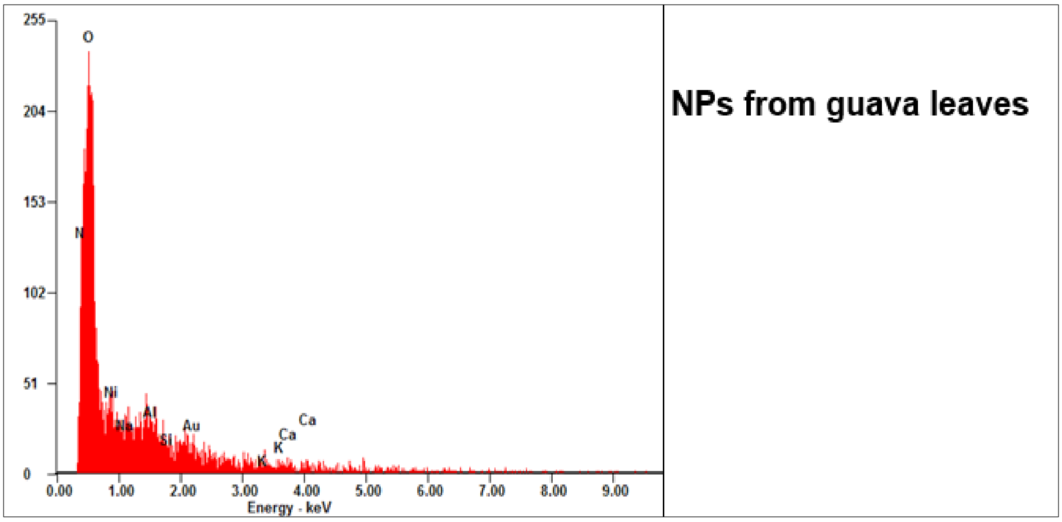

Fig 6: EDAX for NiO NPs from guava leaves

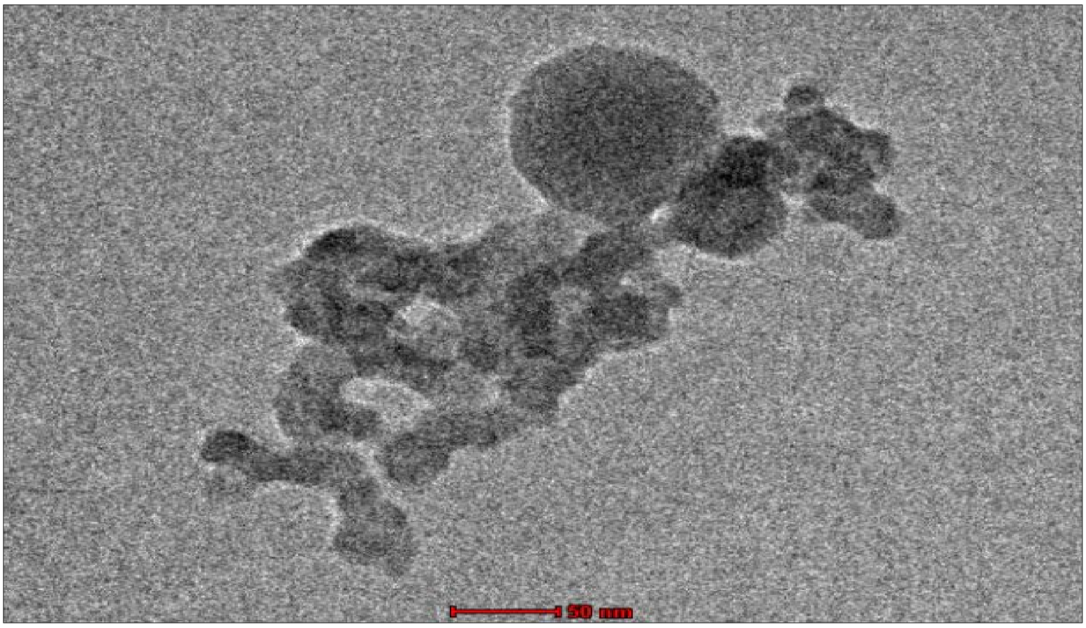

Fig 7: TEM for guava leaves

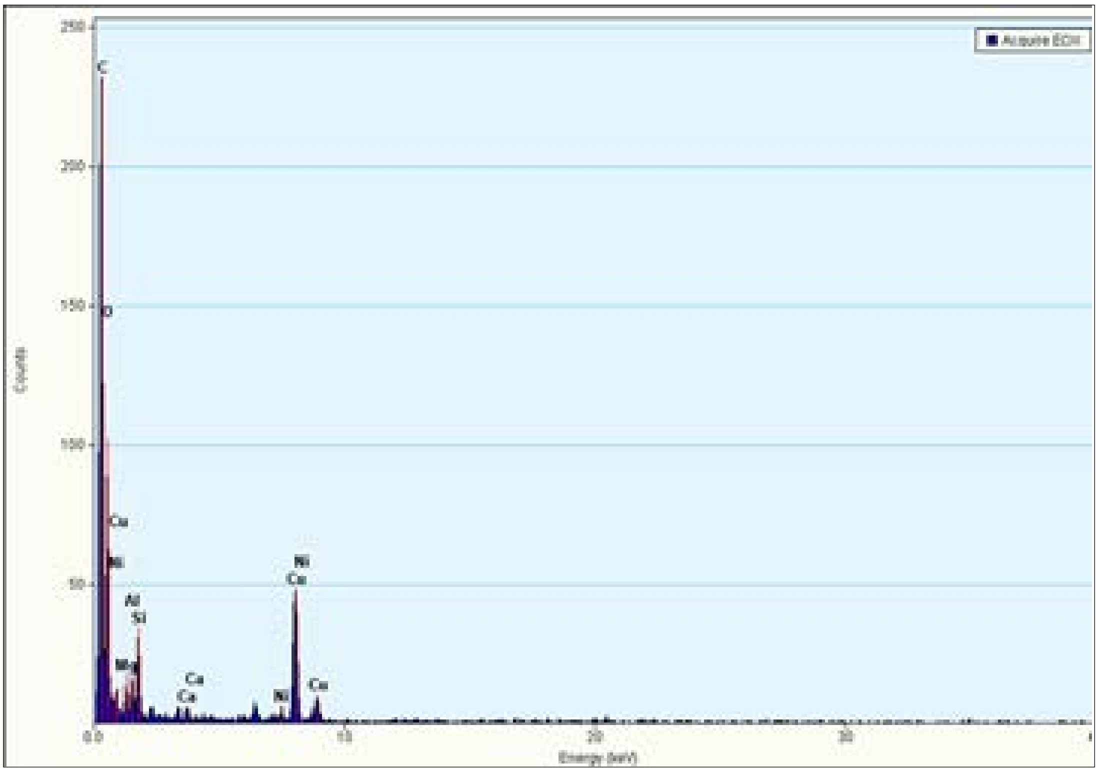

Fig 8: EDAX for guava leaves

4. Conclusion

In this study, NiO NPs was synthesized by *Psidium guajava* leaves aqueous extract. UV-Visible spectrum showed a distinct peak around 330 nm, which is specific for NiO NPs. SEM analysis confirmed that the highly agglomerated spherically shape of the synthesized nickel oxide nano-particles. The TEM images indicated that Ni NPs had a homogeneously spherical shape and found to support the size reduction and confirmed that the synthesized nanoparticles are in nanometer range. EDX results confirmed the presence of nickel and oxygen in the synthesized NiO NPs. FT-IR studies clearly showed the formation of NiO and indicated that the plant extract contains various phytochemicals, which work as capping and stabilizing agent for the synthesized NiO NPs. From the analyses of results, it is clear that the precursors have played a vital role in surface morphology and structure of NiO NPs. Our results confirm the potential of *Psidium guajava* for the synthesis of NiO NPs in a simple, fast and ecofriendly way.

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