

Development and Characterization of Local Content Catalyst
(Montmorillonite Clay) Supported Nickel OxideMUREGI, Muhammad Abubakar^{1*}, M. S. Abolarin¹, O. J. Okegbile¹, and Elizabeth J. Eterigho²¹Department of Mechanical Engineering, School of Infrastructure, Process Engineering and Technology, Federal University of Technology²Department of Chemical Engineering, School of Infrastructure, Process Engineering and Technology, Federal University of Technology, Minna, Niger State, Nigeria.* Corresponding author: muregi.pg718024@st.futminna.edu.ng**Abstract:**

In this study, natural Montmorillonite clay was modified with Nickle Oxide at seven different ratios, 1:1 to 1:4 of Nickle oxide to Montmorillonite clay and the reverse. The modified catalysts were calcined at 800°C for a period of four hours and characterized for its catalytic properties. The modifications of the montmorillonite clay were performed intending to obtain catalysts with different textural and catalytic properties that could improve the reaction under study; bioethanol production. The modified clays were obtained by direct homogenous mixing of both clay and Nickle oxide before calcination. Characterization analysis of the seven samples including raw clay and nickel oxide were conducted. The resulted calcined catalysts were subjected to characterization viz-a-viz BET, SEM and EDX. The resulted catalyst was found to have an increased surface area compared to either that of montmorillonite clay or Nickle oxide. The catalysts with the higher surface area of 498m²/g was found to be that of 1:4 ratio of Nickle Oxide to montmorillonite clay respectively for both the calcined and modified.

Keywords: Natural, Montmorillonite clay, Nickle Oxide, calcination, catalytic properties

Introduction

The limit of fossil fuel reserve is quite clear and the concern of global warming is growing, petroleum which will undoubtedly deplete, since it is a non-renewable resource. It is estimated that crude oil, natural gas and coal will decline worldwide and will drastically reduce/extinct in the next fifty years (Hung *et al.*, 2008). Sequel to this and evidence from literature, it is estimated that over 98% of carbon emission comes from combustion or burning of fossil fuel. Demirbas (2009). Biofuels provide a way of shifting to low-carbon, non-petroleum fuels, either for blending to increase octane rating and/or reduce carbon dioxide (CO₂) emission or replaced completely as automotive fuel. Balat(2011) Carbon dioxide (CO₂) being the primary gas that causes global warming (Hadjipaschalis *et al.*, 2009). Ethanol fuel (ethyl alcohol) is a clear burning alternative fuel made from feedstock (corn, cassava, sugar cane, vegetable matters, animal fats, to mention but a few) it is oxygenated (high oxygen content) with high octane rating and naturally burns. It is

renewable and a sustainable energy of organic chemical compound comprising of hydrogen, carbon and oxygen.

However, for related reasons, ethanol combustion also reacts more with atmospheric nitrogen, which can marginally increase emissions of ozone-forming nitrogen oxide (NO₂) gases. It also contains a negligible amount of sulphur compared to petroleum, blending ethanol with gasoline helps to reduce the sulphur content. However, even at high concentrations of ethanol, minimal amounts of water will draw the ethanol out of the blend away from the gasoline. Ethanol and gasoline are very similar in specific gravity. The two fuels mix readily with minimal agitation, since the biomass used to produce ethanol is created by photosynthesis and the carbon dioxide formed by the combustion of ethanol is recycled back.. The net reduction in greenhouse gases related to ethanol's displacement of petroleum fuel can vary substantially depending upon the amount of fossil fuel used in the ethanol fuel blend process. Clay is

the most primitive and commonest type of sand found on earth surface. (Montmorillonite clay) used for production of catalyst, clays generally constitutes minerals that are hydrous silicates of aluminum, magnesium, with significant amounts of iron, nickel, chromium and other elements. In the crystalline structure as an isomorphic substitution, the peculiar crystalline structure of some clay minerals generates a capacity of reversible exchange with organic or inorganic elements and metal-organic, the exchange of such elements can generate active centers in the clay minerals, making them catalysts, which can be used for a large number of chemical reactions in industries. **Montmorillonite** is a very soft phyllosilicate group of minerals that typically form in microscopic crystals forming clay that is a member of the smectite group of 2:1 clay, meaning that it has two tetrahedral sheets sandwiching a central octahedral sheet. The particles are plate-shaped with an average diameter of approximately one micrometer.

The function of the catalyst support is to enable the formation of large particle table top of catalyst composed of every small readily sinter able crystals of catalyst which are prevented from coalescing by been separated from each other by the support component. The support component clearly as to be more resistant to sintering then the catalytic species. Mostly supports are applied because they combine a relatively high dispersion (amount of active surface) with high degree of thermo-stability of catalytic component. The preparation of supported catalyst align all unit operations towards dispersing an active agent on a support that may inert or catalytically active. The wetting of support with solution or slurry of active phase precursor in the operation the characterized such a preparation (Moronta *et al.*, 2005).

Experimental

The montmorillonite clay used for production of local content catalyst was pulverized into particles, sieved into powdered form. 175g of Nickel oxide and pulverized montmorillonite clay were weighed respectively on Medifield electronic balance of

0.00 sensitivity. 5ml of de-ionize water was measured in the sample bottle for blending the two samples (montmorillonite clay and nickel oxide) to be used for production of local catalyst. Nickel oxide was subjected to solubility test and its hygroscopic nature test in air before blending was commenced. A number of seven different samples were prepared in crucibles base on specified ratios (1:1, 1:2, 1:3, 1:4, 4:1, 3:1, and 2:1) respectively, the samples were blend with de-ionized water and stirred continually until it appears in jelly form, then dried in the furnace. The seven samples were arranged in the oven for calcination, the oven was "ON" and set at a temperature of 800°C for a period of four hours, after the calcination period the oven was switched "OFF" and left closed living the samples inside the oven to allow for natural cooling over night, after which the samples were removed from the oven and re-weighed. "Calcination is a form of heat treatment in absence of oxygen, over a specified number of hours at a recommended temperature below the melting point of the samples to remove moisture and volatile contents thereby increasing the performance of the catalyst". The sample were reweighed and recorded loss of 59.29g to moistures and other volatile impurities, with a final weight of 290.71g as against the initial weight of 350g

Characterization of the as-synthesized catalyst.

Characterization analysis of the seven samples including raw clay and nickel oxide were conducted on sixteen using Brunauer, Emmett and Teller (BET) to determine their respective surface area and pore size. Each sample were properly weighed in the cell bulb into the heating mantle and clamped so that the sample cell is held firmly, then insert into preparation station, outgassing was initiated to a temperature of 300 °C for a period of 3 hours, and the instrument was then turned "OFF" to allow the sample to cool. The outgas station was then unload to remove the sample and reweighed to determine the post outgases.

Results and Discussion

Figures 1 shows the result of the surface areas of modified clay of seven different samples (1:1, 1:2, 1:3, 1:4, 2:1, 3:1 and 4:1) as determined by BET analysis. This shows that the higher the nickel oxide the larger the surface area and pore size (Sharkawy 2013).

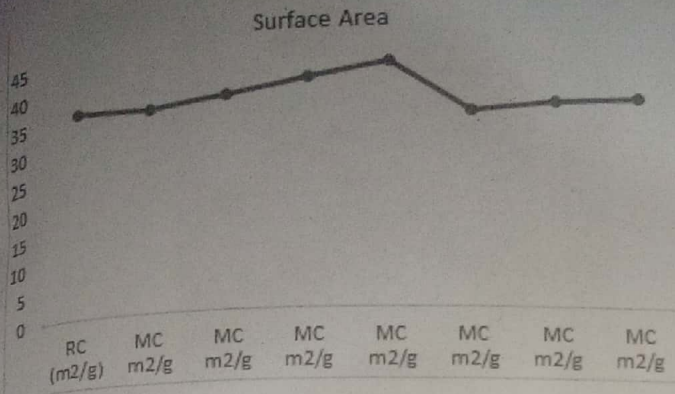


Figure 1: BET analysis of seven modified Montmorillonite clay with Nickle oxide samples (1:1, 1:2, 1:3, 1:4, 2:1, 3:1 and 4:1)

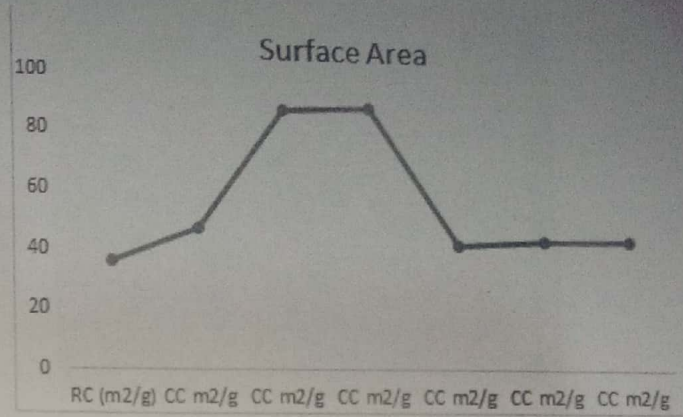
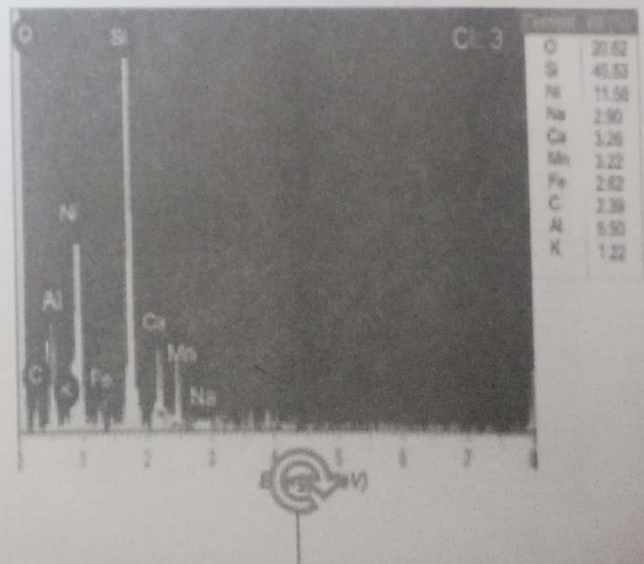
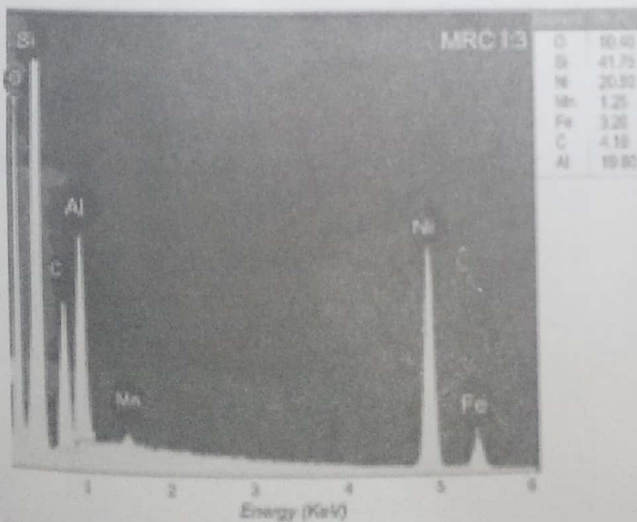
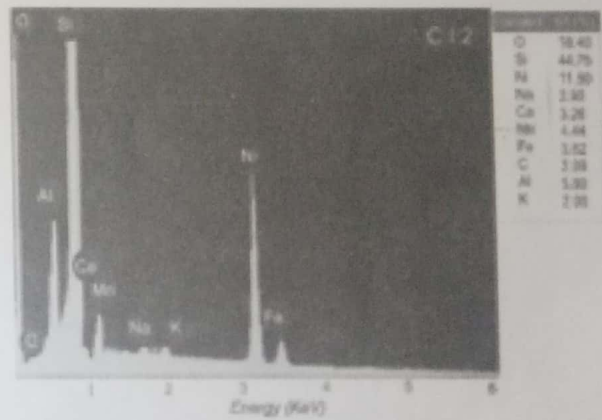
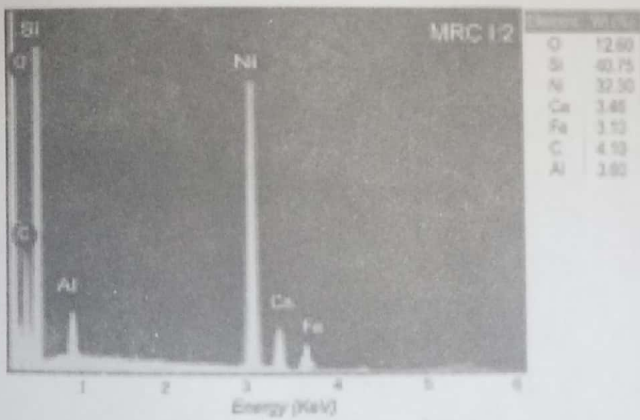


Figure 2: The surface area of calcined samples (1:1, 1:2, 1:3, 1:4, 2:1, 3:1 and 4:1)

Figure 2 represents the result of surface area of the calcined samples (1:1, 1:2, 1:3, 1:4, 2:1, 3:1 and 4:1) as determined from BET analysis). The results indicated higher surface areas for calcined samples compared to the raw mixed samples. This implies that calcination of the samples has a remarkably effect on the morphology of the samples.

The EDX analysis showed the various elemental composition of the different ratios of the formulation. From the spectra, the presence of SiO₂ confirms that the clay is Montmorillonite with the presence of NiO₂ and other element present in the clay. Calcination has effect on both the Montmorillonite clay and the Nickle Oxide.



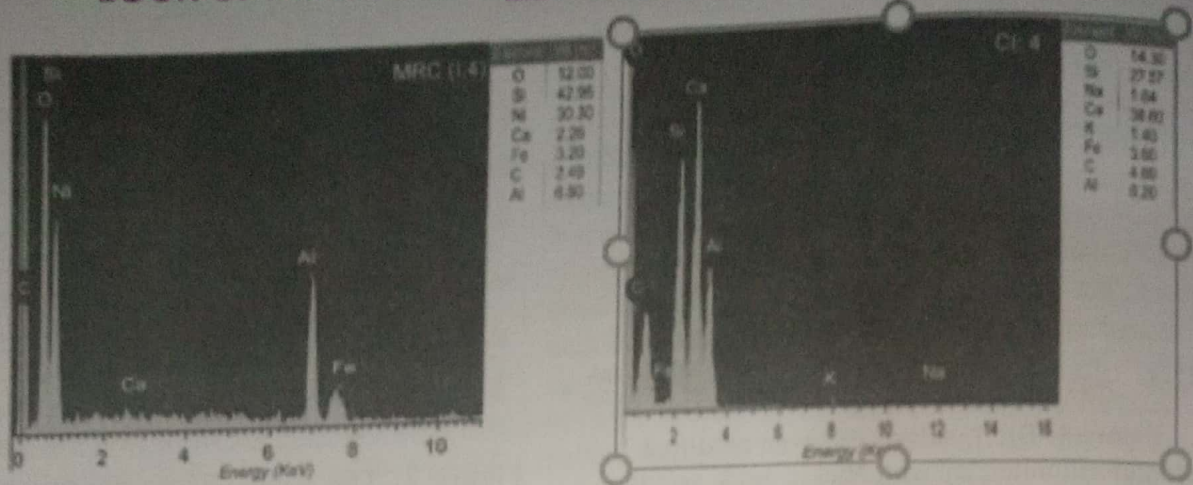


Figure 3: the EDX results of modified clay with its calcined form in different ratios (1:2, 1:3, 1:4)

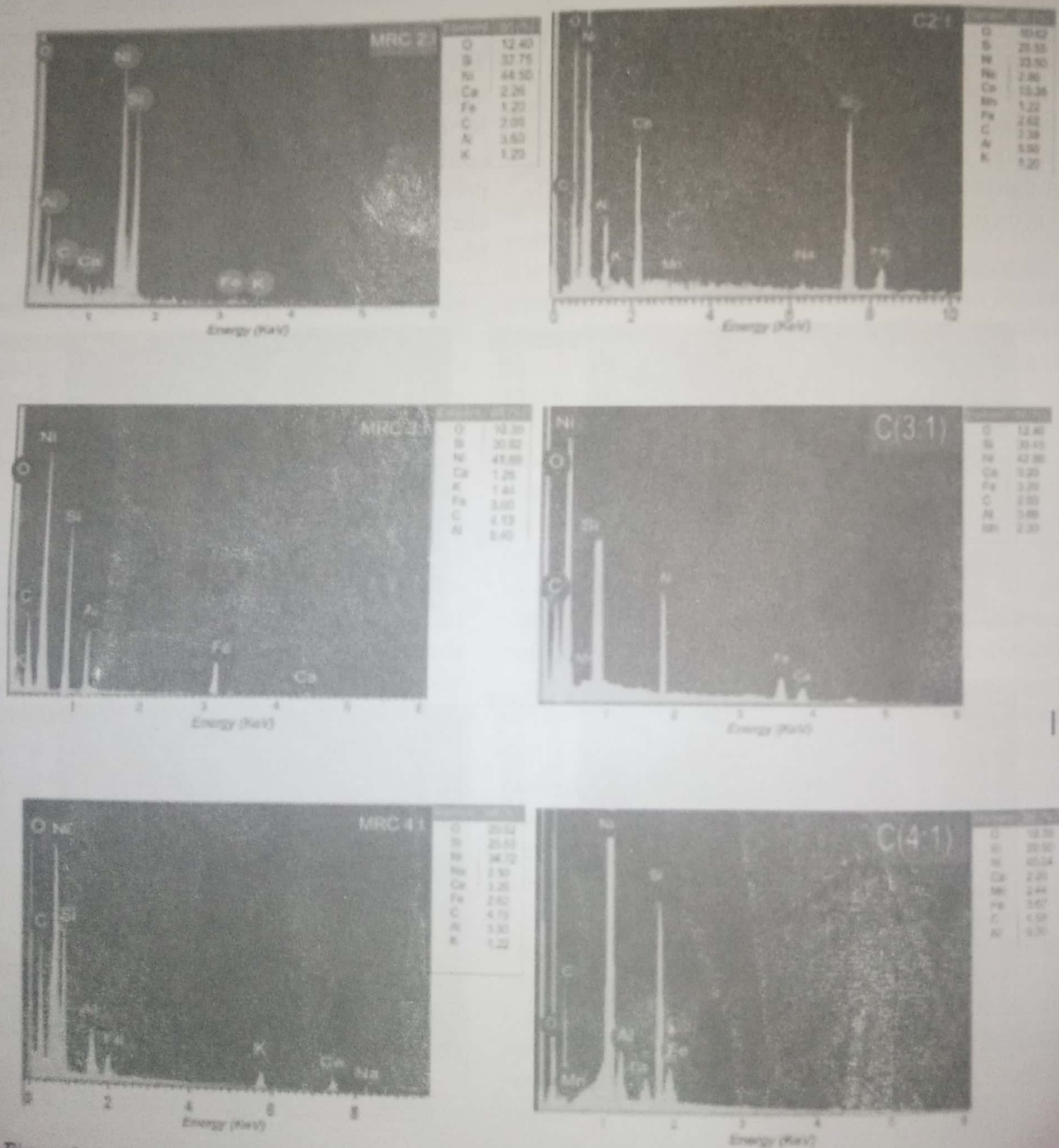


Figure 4: the EDX results of modified clay with its calcined form in different ratios (2:1, 3:1, 4:1)

The results of the FTIR revealed similar trend as discussed in both cases of BET and EDX. The calcination has tremendous effect on the O-H functional group (Ajiboye et al., 2012)

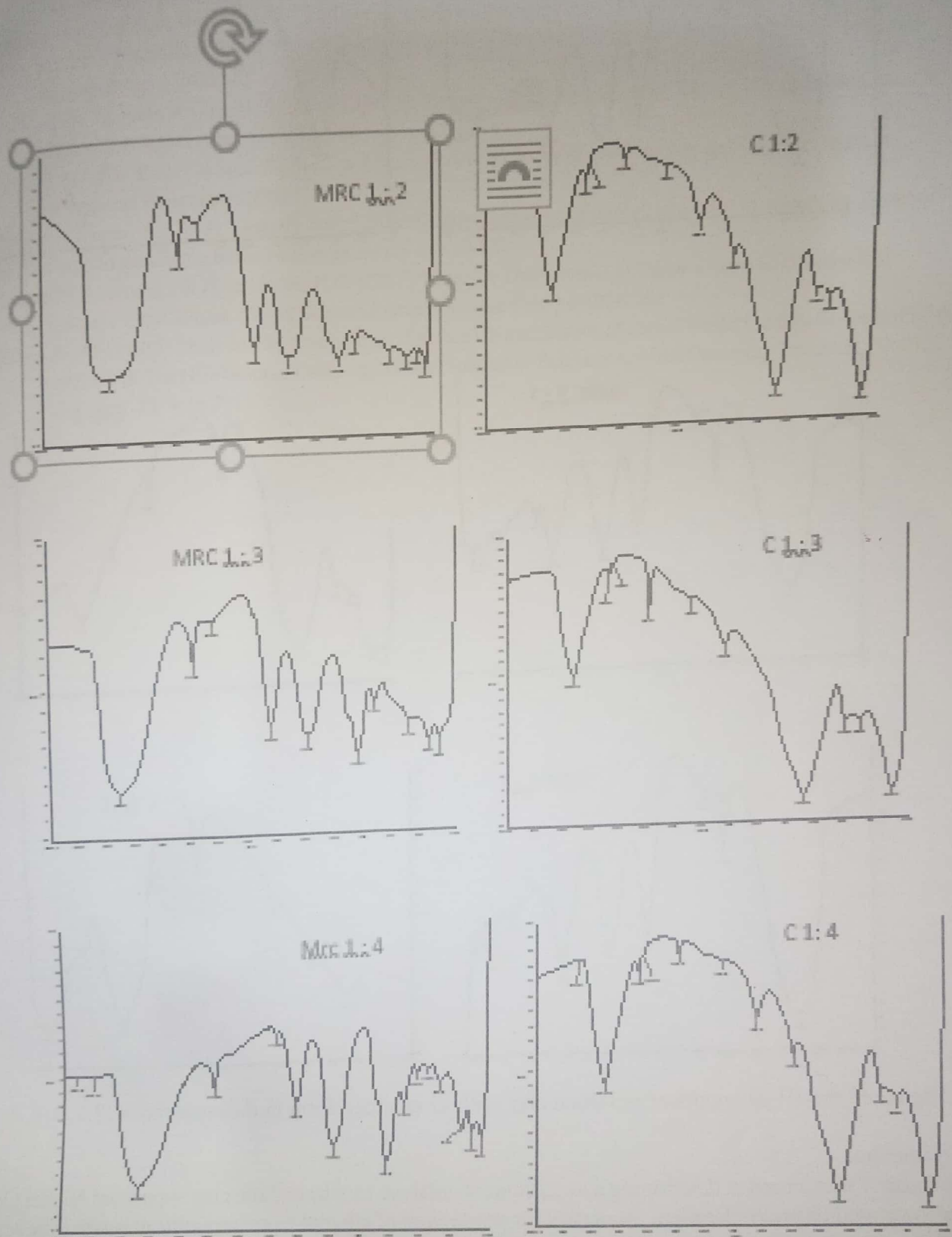


Figure 5: the FTIR results of modified clay with its calcined forms in different ratios (1:2, 1:3, 1:4)

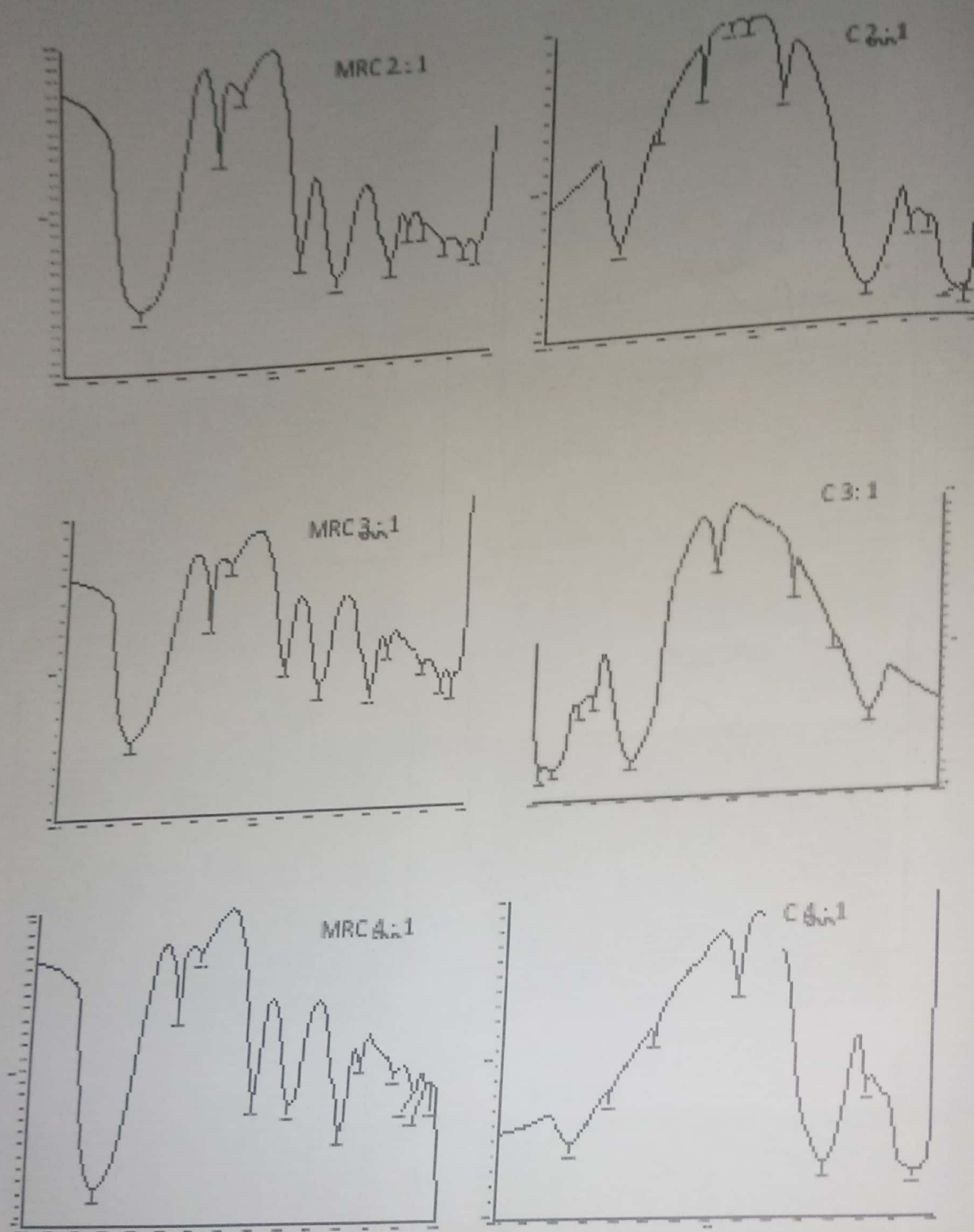


Figure 6: the FTIR results of modified clay with its calcined form in different ratios (2:1, 3:1, 4:1)

Conclusion

This paper was aimed at developing a local content catalyst; montmorillonite clay supported Nickel Oxide for improved physico-chemical properties for production of ethanol from agricultural waste. The results obtained from the present investigations are of importance as the choice of promoter suitable to support nickel is based on theoretical and experimental aspects. Surface characteristics of the prepared catalysts including the specific surface area, Pore volume and radius were performed, these showed that as the amount of nickel to the clay varies progressively, the surface area increased until after 1:3 ratio.

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