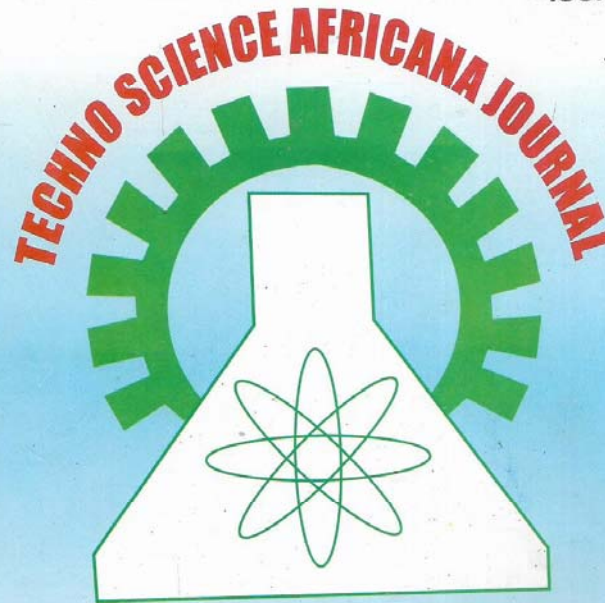




Umar Ahmadu
Physics Concept - Feyn

Vol. 4 Number 1 Dec., 2009
TECHNO SCIENCE AFRICANA JOURNAL

ISSN 2006-2273



**Kano University Of Science &
Technology, Wudil**
P. M. B. 3244, Kano.

UMAR AHMADU PHYSICS DONE

email: africanajournal@yahoo.com

TECHNO SCIENCE AFRICANA JOURNAL

TECHNO SCIENCE AFRICANA JOURNAL

MISSION: The Techno science Africana journal is aimed at facilitating scientific research and promoting academic excellence. It also serves as an avenue for stimulating academic competition among staff.

VISION: Techno Science Africana Journal to become a world-class Journal in the field of science and technology.

FREQUENCY: Techno Science Africana is a biannual Journal, published in June and December every year.

SCOPE: Techno Science Africana Journal is a peer-reviewed journal that publishes original research work, scientific papers and technical reports in all the field of science and technology (Pure science, Agriculture, Engineering Environmental science and related field.)

STRUCTURE: The Journal language is English and all contribution should be submitted in English. It publishes original quality articles which are reporting advances in theory, techniques methodology applications and practice, general survey and critical reviews, etc.

MANAGERIAL STRUCTURE: The Editorial board is made up of the following:

1. EDITORIAL BOARD

Dr. Magashi Auwal Ibrahim	Editor-in-chief
Dr. Musa Tukur Yakasai	Member
Dr. Babangida Sani	Member
Dr. Umar Adamu	Member
Arc. Ahmad Sagab Sani	Member
Eng. Atiku Ahmad	Member
Alhaji Sabo Nayaya	Member
Alhaji Mukhtar Aminu	Secretary

2. ASSOCIATE EDITORS

Dr. Mohammed Abdu Wailare
Dr. Lawan Danlarai Fagwalawa
M. Sani Mohammad Yahaya

TECHNO SCIENCE AFRICANA JOURNAL

TECHNO
SCIENCE
AFRICANA
JOURNAL

ADVISORY BOARD MEMBERS

Prof. I.S. Bushwat (Animal scie.-ATBU)
Prof. A. Bindawa (Agronomy-BUK)
Prof. R.M. Sani (Agric. Econs-ATBU)
Prof. Zanzan Uji Akaka (Arch.-ABU)
Prof. Bashir Garba (chemistry-Energy Commission, Abuja)
Prof. T.K. Atala (Chemistry-)
Prof. Muhammad Yahuza Bello (Computer-BUK)
Prof. U.G. Dambatta (Mech.Eng.-BUK)
Prof. Labo Papoola (Forestry-UI)
Prof. Kabir Ahmed (Geography-BUK)
Prof. E. A. Olafin (Geography-BUK)
Prof. M. S. Audu (Maths-FUT Minna)
Prof. Hari-Hara (Physics-ABU)
Prof. A.O. Musa (Physics- BUK)
Prof A.A. Voh (Jnr) (ABU Zaria)
Prof. A.I. Saminu (Soil Science-ATBU)
Dr. S.S. Noma (Soil Science-UDUS)

TABLE OF CONTENTS

THE EFFECTS OF INTERCROPPING WHEAT WITH FABA BEAN ON WHEAT GRAIN QUALITY Daraja, Y.B.	1 - 6
EFFECT OF COMBINED APPLICATION OF FARMYARD MANURE (FYM) AND NPK ON GRAIN YIELD OF OPEN POLLINATED MAIZE (ZEA MAYS L.) Jakusko, B.B. and Fakuta, N.M.	7 - 9
FUNGAL DETERIORATION OF DRIED YAM CHIPS ON SALE IN TWO SELECTED MARKETS IN KANO STATE - NIGERIA Yahaya, S. M. and Fatima, A.	10 - 12
STEADY STATE MODEL OF AN INDUCTION GENERATOR USING SIMULINK Ben Gonoh and Mahmoud L. Sabo	13 - 18
WIND ENERGY POTENTIAL FOR ELECTRIC POWER GENERATION IN SOME NORTHERN STATES OF NIGERIA S. B. Ibrahim	19 - 25
THE EFFECT OF POWER FACTOR ON LOAD FLOW ANALYSIS: A CASE STUDY OF PHCN, DOKA BUSINESS UNIT, KADUNA Benjamin Akuso Gonoh and Zakari, Monday Adejoh -	26 - 31
RESEARCH AND DEVELOPMENT IN NIGERIA: A SURVEY OF FACILITIES, PROBLEMS AND INSTITUTIONAL ISSUES IN THE SYNTHESIS AND CHARACTERIZATION OF SODIUM SUPERIONIC CONDUCTOR NASICON, Na, Li, Zr (PO) ₃ Ahmadu U., Musa, A.O., Jonah, S.A. and Isah, K.U.	32 - 40
DYNAMIC MODEL OF AN INDUCTION GENERATOR WITH INVERTER EXCITATION USING SIMULINK Ben Gonoh and Mahmoud L. Sabo	41 - 47
DETERMINATION OF PARAMETERS FOR COMBINING GRINDING AND SURFACE HARDENING OF CRANKSHAFT DURING REPAIR OPERATIONS Mahdi Makoyo	48 - 53
DEVELOPMENT OF CODES FOR GROUP TECHNOLOGY MANUFACTURING OF SOME SELECTED MARK II BORE HOLE PUMP PARTS M. S. Dambatta and Mahdi Makoyo	54 - 61
IMPLEMENTATION OF A SIX-LEGGED ROBOT USING PIC16F84 Sani Gaya M., M. Bashir, Bashir M.Sa'ad	62 - 69
GREENHOUSE EFFECT AND GLOBAL WARMING: THE ANTHROPOGENIC SOURCES OF MAJOR GREENHOUSE GAS T. S. Bichi	70 - 73
FACTORS INFLUENCING THE OCCURRENCE OF MALARIA INFECTION IN KANO MUNICIPAL LOCAL GOVERNMENT AREA Tukur, A. I.	74 - 79
AN APPRAISAL OF THE RIGHTS OF PASTORALIST TO GRAZING FIELDS, CASE STUDY OF NORTHEAST OF ADAMAWA STATE Musa Dalil	80 - 83

SUITABILITY OF GROUNDWATER FOR VARIOUS USES IN KABO AREA, KANO STATE Garba Iliyasu	84 - 90
PHYSICO-CHEMICAL ASSESSMENT OF SOME PROCESSED YOGHURT SOLD IN KANO METROPOLIS, KANO-NIGERIA Shehu, A.A.	91 - 99
RAINWATER HARVESTING FOR DOMESTIC WATER NEEDS IN KANO METROPOLIS Aliyu Baba Nabegu	100 - 105
NEURAL NETWORK CONTROL OF A MAGNETIC LEVITATION SYSTEM Mustapha Muhammad and B. A. Gonoh	106 - 115
RELIABILITY ASSESSMENT OF POWER DISTRIBUTION OF SIX 33kV INDUSTRIAL FEEDERS IN KANO STATE, NIGERIA S. S. Adamu and Nuhu Aminu	116 - 125
NONLINEAR MODEL OF A MAGNETIC LEVITATION SYSTEM Mustapha Muhammad and B. A. Gonoh	126 - 133
TRANSIENT SIMULATION OF INDUCTION MACHINE USING CONVENTIONAL MODEL S.B. Ibrahim and B.A. Gonoh	134 - 139
CONVENTIONAL MODELLING OF INDUCTION MACHINE - A DQ MODEL APPROACH S.B. Ibrahim and B.A. Gonoh	140 - 150
THE PERCEPTION AND BELIEF ABOUT HIV/AIDS IN WUDIL LOCAL COMMUNITY, KANO, NIGERIA Adamu Mustapha	151 - 155
ECONOMIC ANALYSIS OF POULTRY PRODUCTION IN KUMBOTSO LOCAL GOVERNMENT AREA, KANO STATE- NIGERIA Yakasai, M. T.	156 - 161
CROSS CORRELATION ANALYSIS AS A TOOL FOR STRUCTURAL TREND DEDUCTION: A CASE STUDY OF GONGOLA Abubakar Y. I.	162 - 170
ECONOMICS OF GUM ARABIC (ACACIA SPP) PRODUCTION: A CASE STUDY OF YOBE STATE, NIGERIA Yakasai, M.T.	171 - 177
INCIDENCE OF <i>SALMONELLA</i> SPECIE AMONGST PATIENTS ATTENDING WUDIL GENERAL HOSPITAL, KANO STATE, NIGERIA Ahmed, I. and Aminu, I.	178 - 181
AN ASSESSMENT OF TOXICITY OF SOME METALS ALONG RIVER JAKARA IN URBAN KANO AND THEIR HEALTH IMPLICATIONS Bichi, A.A., Iguisi, E.O., and Bello, A.L.	182 - 187

RESEARCH AND DEVELOPMENT IN NIGERIA: A SURVEY OF FACILITIES, PROBLEMS AND INSTITUTIONAL ISSUES IN THE SYNTHESIS AND CHARACTERIZATION OF SODIUM SUPERIONIC CONDUCTOR NASICON, $\text{Na}_{1-x}\text{Li}_x\text{Zr}_2(\text{PO}_4)_3$

*Ahmadu U., **Musa, A.O., ***Jonah, S.A. and *Isah, K.U.

*Department of Physics, Federal University of Technology, Minna

**Department of Physics, Bayero University, Kano

***Centre for Energy Research and Training, Ahmadu Bello University, Zaria

*Corresponding author: u.ahmadu@yahoo.com

ABSTRACT

The importance of materials synthesis and characterization for the purpose of tailoring them for specific applications which could potentially assuage the various problems faced by and affecting our bludgeoning economy cannot be over emphasized, particularly in terms of identifying and developing materials that could have the potential to complement our dependence on fossil fuels and thus alleviate the recurrent and persistent fuel crises in the energy sector. Moreover, there is enormous research activity taking place in various fields to develop alternative and renewable sources of energy with enormous implications for our revenue base. The study of NASICON materials for energy applications in diverse fields such as fuel cells for transportation, biomaterials, nuclear waste demobilization and environmental concerns, among others, has been actively pursued in recent times. However, the effort required to synthesize and characterize these materials locally is enormously daunting in terms of information, chemicals, facilities, experts, constant power supply and unfavourable institutional framework suitable for the successful implementation of research. This paper explores and highlights these problems based on an ongoing research on Sodium Zirconium Phosphate (NZP), a member of the NASICON family and proffers solutions to them.

Keywords: Superionic conductors; NASICON; Sodium Zirconium Phosphate; Solid state synthesis

INTRODUCTION

Undertaking research in Nigeria for academic purposes that reflect basic science and technology or for applied purposes is a herculean task which can blunt the doggedness of even the most determined researcher. Different kinds of problems are faced by researchers in different fields, just as the issues at stake in the various fields may be walls apart. There are problems that remain inherently the same, nevertheless. However, in the area of materials synthesis and characterization, the challenges are daunting and can overwhelm even the most confident and ambitious researcher.

In the academics, research and publications are part and parcel of the nerve of the institutions, but lack of identification and recognition of the peculiar problems affecting the output of research and the training needs in the different disciplines and the subsequent enactment of strategic policy to address them has lead to researchers in specific disciplines being disadvantaged in terms of the ease with which they could carry out research and publications. This has slowed down the pace of their progress through the academic ladder and curtailed their potential academic output for reasons not due to default on their part, rather institutional failure to address the peculiar problems that exist in the field. The challenges are multifaceted in terms of facilities, materials, funding and even general information relevant to a well-coordinated and targeted research which would have involved minimal effort. It is not

claimed here that the problems are entirely exclusive to this research rather that they are more pronounced and something need to be done about it. The paper is based on an ongoing work by the authors in the course of conducting research on a family member of ionic conductor NASICON (Na-Super-Ionic-CONductor), i.e., Sodium Zirconium Phosphate, NZP. Superionic conductors, also known as solid electrolytes, are a class of materials exhibiting high ionic conductivity at elevated temperatures. They may be jelly, rubbery and solid in nature and 1-, 2- or 3-dimensional in structure. Their conductivity may reach 10^{-1} - 10^3Sm^{-1} or 10^{-2} - 10^5Scm^{-1} (Yip, 2005) at room temperatures and are insulators. The interest in NASICON is due to their three-dimensional structure which offers increased ionic conductivity suitable for electrochemical applications, the flexibility with which some of the ions could be substituted and thus lead to different physical and chemical properties and potential applications for the ensuing compounds, such as in immobilization of nuclear wastes by NZP(Kutukcu, 2005), environmental gas sensors, electrochromic devices for displays, rechargeable lithium ion batteries for laptop computers and mobile phones and Solid Oxide Fuel Cells (SOFCs) for transportation; others include thermal insulation for diesel engine exhaust components, space technology optical systems, substrate material for electronic packaging, braze fixtures for aircraft engines (Tantri et al., 2000), among others.

Former U.S. Presidential candidate, John McCain did promise any researcher or group able to develop fuel cells, 300 million Dollars (Tantri et al, 2000). This came on the heels of the recent fuel crises that engulfed the World and that saw the prize of petrol reaching 140 US Dollars, a record price. SOFCs can be used in the area of transportation to power vehicles and thus complement the use of fossil fuels. The problems and challenges in the study of these advanced materials include the need to improve their conductivity at room temperature; reduce the temperature to near ambient and understanding the scientific basis of their conductivity and to tailor them for appropriate technological applications by changing their composition and structure. Many studies have been carried out on these materials to fully characterize them for appropriate applications, such as structural, thermal, dielectric properties, impedance spectroscopy at different frequencies and temperatures, ionic conductivity at different temperatures, Fourier Transform Infrared spectroscopy (FTIR), among others.

The objectives of the paper are therefore to present to researchers the relevance and wide range of applications of this class of materials, their properties and characterization techniques and highlight the problems faced in their characterization for prospective researchers. Also to generally identify with the problems of researchers which are of diverse perspectives and intensity so that solutions can be proffered to improve the situation. The paper is expected to induce the will to formulate policies that will improve the research atmosphere, considering the multifaceted nature of the problems raised in our various institutions to stop them being run as government bureaucracies by administrators and policy formulators at those levels. *Institutions and centres*, wherever mentioned refer to dedicated research centres, agencies, etc., in particular and Universities in general, unless otherwise stated.

NASICON and Sodium Zirconium Phosphate (NZP) Structure and Properties

Sodium Superionic conductor (NASICON), basically inorganic ceramic material, has attracted scientific and technological attention due to its promising electrical properties suitable for gas sensors applications and energy storage systems (Aono et al, 2002; Kida et al, 2001; Travers et al, 2002). It has some useful and unique characteristics such as low sintering temperature, three-dimensional framework and considerably good ionic conductivity compared to other solid electrolytes particularly alumina (Hong, 1976; Gordon et al, 1981). The general formula of these compounds is represented as $\text{Na}_{x-3}\text{Zr}_x\text{Si}_x\text{P}_3\text{O}_{12}$ ($x=0-3$). It is a three-dimensional network of ZrO_6 octahedra sharing corners with PO_4/SiO_4 tetrahedra. The Na⁺ are located in the interstitial sites in the framework and ionic conduction takes place when Sodium ions move

from one site to another through bottlenecks formed by oxygen ions (Dong-mei et al, 2007). It has a strong covalent bond. The total electrical conductivity strongly depends on the density and nature of the grain boundaries (Fuentes et al, 2001; Shimizu et al, 2000). NASICON compounds exhibit a few crystalline forms depending on temperature and composition (x) and most sintered NASICON contain certain amount of ZrO_2 second phases. NASICONs are usually synthesized by two methods, the traditional ceramic route or sol-gel method. Only the experimental details of the solid state synthesis are provided here. The microstructure of samples is usually affected by the preparation methods (Ahmad et al, 1987; Schaf et al, 2004).

The structural formula of NZP is $[\text{M}^+][\text{M}^{2+}][\text{A}_2^{IV}][\text{B}_2^VI]\text{O}_{12}$, where M^+ , M^{2+} are interstitial sites occupied partially or fully by Na or other substituting ions. A and B lattice sites are primarily occupied by Zr and P or by possible substituting ions. The crystallographic structure of the parent composition, $\text{NaZr}_2(\text{PO}_4)_3$, was first determined by Hangman and Kriekgaard in 1968 (Breval et al, 1995). The above general formula for NASICONs are indeed solid solutions derived from $\text{NaZr}_2(\text{PO}_4)_3$ by partial replacement of P by Si with Na excess to balance the negatively charged framework (Mouahid et al, 2001). NZP materials generally exhibit anisotropic expansion properties, as thermal expansion behavior may involve the expansion of some axes and contraction of others (White, 2006). The composition of NZP family being studied in this work is $\text{Na}_{x-3}\text{Li}_x\text{Zr}_x(\text{PO}_4)_3$, $x=0-1$ (0, 0.25, 0.50, 0.75 and 1.00). What is required is to substitute fully the amount of Na in the structure by Li and determine the effect on the structural, thermal and electrical properties of the compositions, with a view to applications in energy storage as electrolytes. Lithium cells have a very important market due to their unmatched properties of high potential, very light weight nature and hence very high energy density properties (Mouahid et al, 2001), accounting for 63% of Worldwide sales of portable batteries (Taracson and Armand, 2001). There is intense research effort in the area of three-dimensional Lithium-based solid electrolytes for all solid lithium batteries. NASICON type materials with lithium cations in the channels are candidates as electrolytes in these cells if the conductivities at room temperatures can be enhanced.

Problems, Challenges and Characterization Methods in NASICON Research

Research Theme and Literature Survey

Ideally research theme must be relevant to and have bearing on national problems that we face, starting from the immediate environment within the walls of a university, local government or any other problem which may be brought to the attention of the relevant research institutions by the government or may be derived from the experience of the researcher.

In order to start research one begins from the library and what we find are outdated books that cannot provide meaningful guide to current problems, where available at all. In spite of the vaunted embrace of information technology by many government agencies and institutions, many libraries do not have institutional subscription for online transactions to give researchers free access to journal materials and other useful resources that will enable them achieve their objectives with minimal dissipation of efforts, time and resources. In many cases there is problem with the functioning and effectiveness of such InfoTech centres, particularly in providing internet services that are easily accessible. The researcher thus pays for the cost of browsing, printing and has to put up with all the inconveniences that characterize internet cafes as currently being run in the country. Thus almost 100% of the literature and other resources relevant to this work were obtained via browsing the Web.

Exhaustive and thorough literature review would eventually lead to the researcher making a decision on the subject or topic of the research without full or detailed information within a reasonable time, this may lead to problems in the future as there is no way to know everything from the beginning. The most identifiable feature in the case of the synthesis and characterization of NASICON, NZP is the final decision that was made about the materials to be investigated. Other aspects of the problem were identification of the appropriate characterization techniques needed to derive relevant results expected from the outcome of the work. Some of these techniques, problems and the information they provide are described below and are not limited to the actual characterizations (to be) used.

X-ray powder diffraction techniques are used for structural, lattice parameter, phase and particle size determination of NZP materials. Various designs of such machines are available with different capabilities, accessories and software (using ICDD). Accessories normally accompany these machines which are normally not expressly stated during literature survey, but can become a major problem during the actual research. The survey revealed that XRD machines are available at NSC Kaduna, EMDI Akure and CERD, OAU, Ile-Ife, with different charges and conditions surrounding their use. The XRD for this work is expected to be carried out at CERD. The information from this characterization is important as it determines whether the relevant NASICON has begun to form through crystallization and the different phases present, that is if impure. It can also be used to determine the size of the particles appropriate for the relevant applications.

Scanning electron microscopy (SEM) machines are used for morphological and microstructure to give a graphical idea of the surface features of these substances. Such

information as the distribution of the constituent compounds forming the sample under investigation are observable, homogeneity, grain boundaries, etc., for both calcined and sintered samples. For this investigation, the constituent compounds, homogeneity and grain boundaries are of profound effect on the impedance characteristics of the samples and thus their distribution. In this case the grain size of different sample compositions can be determined. Generally, the machines can be adapted for solid conductors or insulators with some additional accessories, for example, the use of special paints to cover insulators and automation software such as LINCE, for repetitive and quick computations. SEMs are sometimes used in conjunction with EDX(S) (energy dispersive spectroscopy) to determine the elemental compositions of samples as it also provides a profile of the distribution of elements on the surface of the samples. The survey revealed that only BUK, Kano has this machine but can only be used for *electrically conducting materials* for now. However, the sample under study is an insulator, so alternatives are being sought. Elemental compositions may also be determined by other complimentary methods with some special sample preparations, such as AAS (atomic absorption spectroscopy) and X-ray Fluorescence spectroscopy (XRF), regrettably, these cannot detect Li and Na and are available in many institutions. However, inductively coupled plasma optical absorption/emission spectroscopy (ICP-O/AES) which can detect the metallic constituents Zr and P (Jayswal and Chudasama, 2007), not available locally, and neutron activation analysis (NAA), using NIRR-1 research reactor at CERT Zaria and PIXE or Gamma detector at CERD Ife, could detect the whole range of the elements in the NASICON, though there is often the problem of availability of liquid Nitrogen, a problem that impedes the use of these latter facilities. These options are being considered anyhow since they all attempt to ascertain the purity of the investigated compounds in quantitative and qualitative terms to some minimum level of detection.

Another analysis technique that has been used in this field is Fourier Transform Infrared Spectroscopy (FT-IR), currently available in NARICT, Zaria. The infrared spectrum of these substances provides information on their characteristic absorption or transmittance properties. Water of crystallization or hydroxyl groups present can be revealed. Machines such as Bomen MB series spectrometer and Perkin Elmer 1605 model (Jayswal and Chudasama, 2007; Sreenivasu and Chandramouli, 2000), using KBr wafer and relevant software and accessories are used. Wavenumber range could be 400-4000 cm^{-1} . Impedance spectroscopy (IS) is one of the most important tools used in NASICON studies to characterize their electrical properties.

They studied the composition $Ba_{1.5-x}Sr_xZr_xP_4SiO_{24}$ with $x = 0-1.5$, in steps of 0.25 through solid state reaction method. The result showed that the $x = 0$ sample had a negative α (coefficient of thermal expansion) whereas the other extreme composition with $x = 1.5$ had a positive α . The starting chemicals used were $BaCO_3$, $Sr(NO_3)_2$, ZrO_2 , SiO_2 and $NH_4H_2PO_4$. Stoichiometric mixtures of the compounds were hand mixed and dried at $100^\circ C$, followed by heat treatment at $200^\circ C-3h$, $400^\circ C-3h$, $500^\circ C-6h$. Calcinations were carried out at $900^\circ C-9h$. The calcined product was ground and sintered for 14h at $1400^\circ C$ after pelletizing. The samples sintered at $1400^\circ C$ had densities 90-95% of theoretical values. The thermal expansion behavior was measured by a Variable Differential transformer (LVDT) based dilatometer, from room temperature to $800^\circ C$. In their study of NASICON of composition $Na_3Zr_2Si_2PO_{12}$, Fuentes et al (2004) showed that the formation of NASICON is expected to start from $1000-1100^\circ C$, and that this is strongly enhanced at higher temperatures ($1200-1300^\circ C$). The phase formation, sintering behavior and electrical characteristics of NASICON compounds were also studied by Kang and Cho (1999). They correlated the behavior of two NASICON compositions $Na_{3.2}Zr_{1.3}Si_{2.2}P_{0.8}O_{10.5}$, called the Hong and Von Alphen type NASICON respectively. The result showed that it is essential to choose an appropriate sintering temperature and cooling rate to produce sintered NASICON with less ZrO_2 and higher electrical conductivities and that the cooling rates should not be too slow. Also it was observed that denser sintered bodies are favorable for higher electrical conductivity. Lithium compounds (analytical grade) have also been difficult to obtain and are very expensive, such as $LiNO_3$, $LiCl$ (hydrothermal route) and $LiCO_3$. Importation of these chemicals from overseas through the Web also has its peculiar problems, together with other alternatives such as contacting someone overseas. In the efforts at acquiring genuine chemicals one had to contend with attempts to pass off fake or repackaged chemicals for genuine, coupled with the exhaustion of having to travel all over, making phone calls, etc. The salient point to be noted is the lack of foreknowledge of where to get the relevant experts in synthesis and credible sources of genuine chemical suppliers without actually embarking on physical journey which many a time had turned abortive. The main problems faced in this case were in obtaining Furnaces that can function at high temperatures $>1300^\circ C$. Crystallization of NASICON starts above $1000^\circ C$ and reaches its best above $1300^\circ C$. Calcinations and sintering had to be done for various lengths of time, up to 24hours, in some cases. The survey showed that most institutions lack such Furnaces and where available can only go as far as $1200^\circ C$ as permissible limit and 4-10 hours, maximum. In some cases the furnaces have simply broken

down and spare parts are difficult to come by. This does not include the issue of power supply which obviously would not be provided for such a period and would have to be borne by the researcher. High temperature crucibles made of Platinum, Silica, etc. are critical, so that they do not react with the substance under study and are thermally stable. Na and P are volatile at high temperatures. The availability of these crucibles presents their own difficulties.

In the hydrothermal case however, reaction autoclaves are normally used, e. g. acid digestion bombs with inner parts made of PTFE (Polytetrafluoroethylene). PTFE lined digestive bombs are used because Teflon is an inert material so it does not react with the reactants (Ozman, 2004). However, it has a limitation of the size of crystal formed in single crystal experiments which are kept for days at low temperatures, $150-230^\circ C$ (Ozman, 2004). One such digestion bomb was bought for the purpose.

It should be noted that for whatever reason some research institutions simply refuse to enter into any form of communication in respect of applications on the use of their facilities, even though they have it and due process in putting in for such a demand was followed.

Institutional Issues and problems

There are many institutional problems that beset research centres vis-avis the attempt to carry out research in them and thus hamper the effective utilization of the facilities and successful implementation of research to its logical end. Information is not available to an inquirer due to bureaucratic procedures and other nonchalant attitudes toward researchers, in some cases. Many such centers do not operate an open policy towards achieving research objectives and having easy access to information and use of facilities and other resources could be difficult.

The charges that are in place for the use of facilities for characterization or sample preparation are in some cases on the high side. There is need for synergy and communication between research centers and universities in order to arrive at reasonable costs for such analyses for would-be researchers, since most researchers have to pay from their pockets from the meagre resources available to them. A case where a researcher had to pay for pelletization of sample is mind-boggling. There is issue of accommodation for researchers in whichever institution he/she may wish to base research. Where a researcher has plenty of samples and needed different characterization methods and would like to have his/her results immediately and not wait for months on the queue of waiting lists, it is conceivable and reasonable that accommodation is available for such resident researchers, ideally free but reasonable charges might be ok, as against the present manner in which the researcher has to contend with hotel

bills, feeding, local transport, characterization /use of apparatus charges and other miscellaneous expenses. All these and more have to be articulated and harmonized within a strategically formulated and holistic policy framework which embraces all the relevant issues in a workable symbiosis between research centres and universities in particular. The academic or commercial peculiarities or inclination of the research should be reflected in the deliberations in view of the poor funding of academic research in universities and in the country in general. The government in conjunction with research institutions should make funds available and easily accessible for such purposes and in particular seek collaborative initiatives and linkages with overseas institutions in this regard, at least for the experimental phases of research, where such facilities are not locally available. This can greatly improve the situation in the short term.

Another characteristic problem that crisscrosses some of the centres is the potential for a facility to breakdown and no certainty as to when it would be fixed, it could mean the end of the road for a researcher where no other facility is available or it could lead to prolonged period of waiting, more than necessary for the work, since one has to restart the process all over again in the next available laboratory with new charges, etc., that may come into force. In some cases the facility is available and it would be suggested that the scale of the work be compromised, as a safety measure for the upkeep of the facility for fear of breakdown when operated to such capacity as may be required by the researcher, a measure which would not provide the researcher with the needed result in some cases. There is thus the need for a permanent framework to be worked out for the purpose of ensuring local availability of the technical know-how to fix such equipment and possible repository of potential components that could fail in the system. This seem to be the most acute and recurrent problem threatening all institutions with research facilities. It is suggested that these and more should be integrated into the original contract that saw the installation of the equipment in the first place and applicable in all institutions, not only research centres or universities, so that prompt attention can be paid to such facilities within a short notice by the company that supplied and installed the equipment, of course there would be financial implication to this.

Incomplete Systems

One characteristic which feature prominent in some cases where facilities are available is the incomplete nature of some of the facilities in terms of their accessories and other ancillary attachments. This had often rendered many expensive facilities useless and abandoned, simply due to the absence of one component or the other. There is need for inclusiveness of staff working in the relevant field in which the equipment is to be bought to be allowed to fully

participate in all the steps leading to the acquisition of the relevant facility. Present manner where the purchase of equipment excludes experts in the relevant field is counterproductive to our desire to achieve our national objectives through science and technology and even to the spirit and letters of the objectives of such institutions. This problem afflicts virtually all our institutions and its resolution should be pursued in tandem with the training and re-training needs in that field due to the dynamics of technology which results in new and better performing systems on an almost annual basis. This would enable the staff handling the machines to be properly grounded in the theoretical and the experimental nitty-gritty in using such machines and thus maximize the efficient utilization of the facilities towards better accuracy of results. There are cases in which officers manning such machines lack adequate theoretical grounding in the use, data analysis and the technical know-how needed to resolve basic electromechanical problems or similar that could arise in the course of the use of such facilities. Also in some cases the senior officers, of high academic qualifications and with the appropriate theoretical grounding on the subject, who are normally in charge, are rarely available for consultations or show uncooperative attitude to the researcher in solving his problems, by not giving him their time and necessary attention. In many cases the machines have the capability to undertake analyses more extensive in scale than they are hitherto limited to but one would be told that they could not do them, simply because the officer in charge has not done it before or does not know how to do it. There are cases where software that are supposed to be part and parcel of the facilities or certain components thereof are missing and therefore the machines remain only for show. Nevertheless, it must be acknowledged that there are cases in which officers in charge of equipment demonstrate excellent understanding of the theoretical and analytical components of the data derived from such machines, such people should be encouraged.

Prospects for Improvement in Materials Characterization

Nigeria is increasingly creating new research bodies specialized in various fields within universities and outside. There is need to create a database of all the scientific-technological based institutions that we have, with specific reference to their facilities and expertise in various disciplines, their addressees, emails and telephone numbers. These institutions should as matter of urgency publish all their available research facilities, equipment, material, specialized software, chemicals and the list of professionals in the various fields of specializations. The list could be both in print and on the internet and should be frequently updated. They could send copies to all our research institutions.

Similarly, the working state of each equipment or facility should be stated. The idea is that a website for all such institutions where researchers can easily log on to and access virtually all the information he/she requires and not subjected to the problems currently being faced by researchers. The list is not exhaustive and other information deemed relevant could be included, incorporating institutions that deal in materials and have scientific-technological relevance for our research and development purposes.

There is the need for communicating, synergizing and harmonizing, including defining research focus, objectives and problems by these institutions and to pool their resources together to facilitate the establishment of such a database which would be a one stop shop for researchers.

The government should encourage private entrepreneurs to engage in the business of materials characterization so as to make it more affordable and accessible, even as the cost implication might be on the high side for the average researcher, but would make the services easily accessible, creating competition in the process. As a nation there is need to define in a targeted manner, what our problems are and define research objectives accordingly in focused areas to harmonize, strategize and implement research programs that will lead to visible economic dividends, confer respect and recognition on our scientists and engineers. There is no doubt that the present manner in which research is being carried out in most of the sectors and fields and by various intuitions lacks properly defined borders, harmony, target and time-frame-it is open-ended with no target dates for completion or realizing the set objectives and subsequent evaluation needs. There are overlapping, duplicated and diffuse functions. Reorganizing, narrowing down and streamlining of these functions will certainly lead to more effective and better management of scarce resources and effective use of facilities thereby contributing effectively to Nigeria's aspiration for self-reliance, by giving our research at all levels a sense of direction and potency. Though many research institutions have made impact through their consulting services with industries, etc, however, more need to be done to effectively utilize their facilities in a more comprehensive manner. There is need to develop and generate a compendium of all the problems we face in the various sectors of our social, economic, and political lives through defined means, such as workshops, seminars, etc. which should form the nucleus of research by universities and other research centers with targets, evaluation procedures and mechanisms in place to assess achievement and stimulate competition so as to induce the drive necessary to produce results. It will thus make our undergraduate and postgraduate research objectives and goals attuned to our national problems and aspirations

and the output from such endeavors would be visible to Nigerians. It must be noted that at present many Undergraduate to PhD. level research projects, comprising theoretical and construction work with those in research centres, are lying waste, a waste of time, money and energy, due to the fact that works were not based on real problems thrust to the institutions by any government or private institutions in the country and were not generated by any formal process, as observed elsewhere in this work. Thus there is research (R) but no development (D) across all institutions. We must base our research on our national aspirations and these must be generated through workshops and seminars, in particular, and attuned to the vision 2020 of the present Yar'adua administration. For example, energy is the most important problem for now, it is reasonable to redirect 60-70% of research in the country towards this objective from various alternative energy perspectives and the appropriate funding provided. There is presently no such attempt to harmonize our researches with and into the Vision 2020 Programme. The government must encourage or enforce the awarding of contracts of scientific and technically-based problems faced by industries and allied companies to universities and research institutions to solve, without prejudice to their know-how capacities, rather than contracting them out overseas. This will challenge our professionals and researchers and provide them the needed experience and stimulus to proffer solutions to our problems. Our legislators may need to enact a law to enforce such policies to create the enabling environment for our scientific and technological capacity to blossom.

CONCLUSION

The role that Superionic conductors play through NASICON research in fuel cells for applications in transport sector to reduce and diversify our energy base should be explored, as concerted efforts are being exercised all over the World towards realizing this objective. Other relevant applications of these materials in the industry for their tailorability should be researched into. It is obvious that the full characterization of these materials is both extensive in scope and depth and certainly in terms of cost. The present harsh research climate must be subdued in order to ease the problem in terms of increased funding of postgraduate research, accommodation and other miscellaneous expensnses incurred by researchers. The issue of charges for characterization should be addressed synergistically and communicatively between the relevant research-based organizations and universities to energize the present nonexistent communication between these bodies. It is also suggested that the issue of incessant breakdown of equipment, affecting all research-based organizations must be seriously looked into with a view to permanently addressing the problem.

Database development for facilities, manpower, chemicals, expertise and other related matters should be kick-started within an agreed timetable. The need to define research directions in the various sectors together with requisite evaluation mechanisms, time-frame and proper focus on narrow areas must be in our board room for integration with vision 2020. The research in universities and related institutions must be derived from and generated through a national consensus of the problematic areas. All such areas agreed upon as the focus for the next phase, supposedly the Vision 2020 is a programme in phases, must be funded since they are supposedly derived from national problems.

There should be concerted efforts by universities authorities to improve on the provision of facilities for research both at the undergraduate and postgraduate levels and to review the present framework within which fellowships are granted with their attendant implication on the academic progression of staff. Government's research focus annually, biannually, etc., should be gazzeted periodically and according to the peculiar features of the

disciplines. The need to encourage collaborative work in order to minimize wastage and dissipation of energy on projects that are not developed cannot be overemphasized. There should be sources of credible supplies of chemicals certified by the government through its agencies in order not to deceive researchers. The establishment and pursuit of excellence in developing materials characterization facilities is the beginning of the foundation of our economic growth.

Acknowledgements

The authors acknowledge the roles played by Mr. Abiodun Jegede of CERD, OAU, Ife, the Director of the Centre and Mr. Idowu the Secretary, in the use of DTA machine and wish to thank Eng. Shehu Ahmed Isah, Mr. Yemi and Eng. Yakubu of NMDC, Jos, for help with the Furnace work. We are grateful to Dr. Jonathan Yisa, FUT Minna; Prof. Sunday Thomas, Shestco, Abuja and Dr. Habu Nuhu Aliyu, of Chemistry Department, BUK, for their various roles in the chemistry aspect of the work.

REFERENCES

- Ahmad, A.Wheat, T.A., Kuriakose, A.K., Canaday, J.D. and McDonald, A.G. (1987). Dependence of the properties of NASICON on their composition and processing. *Solid State Ionics*, 24:89-97.
- Aono, H., Sadaoka, Y. and Montanaro, L. (2002). Humidity influence on the Co2 response of Potentiometric sensors based on NASICON pellets with new compositions, $\text{Na}_3\text{Zr}_{2(x/4)}\text{Si}_{1-x} \text{P}_x \text{O}_{12}$ ($x=1.333$). *Journal of the American Ceramic Society*, 85(3)585-589.
- Breval, E., Harshe, G. and Agrawal D.K. (1995). Synthesis and Chemical stability of $\text{NaSn}_3\text{P}_3\text{O}_{17}$. *Journal of Material Science Letters*, 14:728-731.
- Dong-mei, Z., Fa, L., Zang-long, X., Wang-Cheng, Z. (2007). Phase Formation and Electrical Characteristics of NASICON Ceramics. *Transactions of nonferrous Metal Society of China*, 17:s1156-s1159.
- Fuentes, R.O., Figueiredo, F.M. and Marques, F.M.B. Processing and electrical properties of Ceramics prepared from yttria-doped Zirconia precursor (2001). *Journal of European Ceramic Society*, 21:737-774.
- Fuentes, R.O., Lamas, D.G., Fernandez, M.E., Rapp, D.E., Figueiredo, F.M., Frade, J.R., Marques, F.M.B. and Franco, J.J. (2004). Restrictions to obtain NASICON by Ceramic route. *Bolletín de la Sociedad Espanola de Ceramica y Vidrio* 14(4):775-779.
- Gordon, R.S., Miller, G.R., McEntire, B.J., Beck, E.D. and Rasmussen, J.R. (1981). Fabrication and characterization of NASICON electrolytes. *Solid State Ionics*, 3/4:243-248.
- Grey, C.P. and Dupre, N. (2004). NMR studies of Cathode Materials for Lithium ion Rechargeable Batteries. *Chemical Review*, 104:4493-4572.
- Hong, H. (1976). Crystal structure and crystal chemical in the system $\text{Na}_3\text{Zr}_2\text{Si}_3\text{P}_x\text{O}_{12}$. *Materials Research Bulletin*, 11:173-182.
- Huang, C-H. Agrawal, D.K., Mickinstry, H.A. and Limaye, S.Y. (1994). Synthesis and Thermal Behaviour of $\text{Ba}_3\text{Zr}_2\text{P}_x\text{Si}_{3-x}\text{O}_{12}$ and $\text{Sr}_3\text{Zr}_2\text{P}_x\text{Si}_{3-x}\text{O}_{12}$. *Journal of Materials Research*, 9(10):2005-2013.
- Jayswal, A. and Chudasama, U. (2007). Synthesis and Characterization of a new Phase of Sodium Zirconium Phosphate for the separation of Metal ions. *Journal of Iranian Chemical Society*, 4(4):510-575.
- Kida, T., Miyachi, Y., Shimano, K. (2001). NASICON thick film based CO_2 sensor prepared by a sol-gel method. *Sensors and Actuators B-Chemical*, 80(1):28-32.
- Kutukcu, M.N. (2005). Synthesis and characterization of Low and negative thermal expansion materials. Unpublished M.Sc. Thesis, School of Chemistry and Biochemistry, Georgia Institute of Technology, Georgia.
- Kumar, P.P. and Yashonath, S. (2006). Ionic Conduction in the Solid state. *Journal of Chemical Science*, 118(1):135-154.
- Kang, H.-B. and Cho, N.H. (1999). Phase Formation, Sintering Behaviour and Electrical Characteristics of NASICON Compounds. *Journal of Materials Science*, 34:5005-5013.

- Mouahid, F.E., Zahir, M., Maldonado-Manso, P., Bruque, S., Losilla, E.R., Aranda, M.A.G., Riuera, A., Leao, C. and Santamaria, J. (2001). Na-Li Exchange of $\text{Na}_{1-x}\text{Ti}_x\text{Al}(\text{PO}_3)_3$ ($0.6 \leq x \leq 0.9$) NASICON Series: a Rietveld and Impedance study. *Journal of Materials Chemistry*, 11:3258-3263.
- Ozmen, B. (2004). Hydrothermal Synthesis of Solid State Materials and Crystallography. Unpublished M.Sc. Thesis, Department of Chemistry, Izmir Institute of Technology, Izmir, Turkey.
- Schaf, O., Weibel, A. and Llewellyn, P. (2004). Preparation and electrical properties of dense Ceramics with NASICON composition sintered at reduced temperatures. *Journal of Electroceramics*, 13(1/3):817-823.
- Shimizu, Y., Ushijima, T. (2000). Sol-Jel processing of NASICON thin film using aqueous complex precursor. *Solid state Ionics*, 132(1/2):143-148.
- Sreenivasu, D. and Chandramouli, V. (2000). Spectroscopic and Transport Studies of Cu^{2+} ion doped in $(40-x)\text{Li}_2\text{O} \cdot x\text{LiF} \cdot 60\text{Bi}_2\text{O}_3$ glasses. *Bulletin of Materials Science*, 23(6):569-573.
- Tantri, P.S., Greetha, K., Umarji, A.M. and Ramasesha, S.K. (2000). Thermal Expansion Behaviour of Barium and Strontium Zirconium Phosphates. *Bulletin of materials Science*, 23(6):491-499.
- Taracson, J.M. and Armand, M. (2001). Issues and Challenges Facing Rechargeable Lithium Batteries. *Nature*, 414:359-367.
- Traversa, E., Muntanaro, L. and Aono, H. (2002). Synthesis of NASICON with new compositions for electrochemical CO_2 sensor. *Journal of Electroceramics*, 5(3):261-272.
- White, K.M. (2006). Low temperature synthesis and characterization of some low positive and negative thermal expansion materials, unpublished Ph.D. Dissertation, department of Chemistry, Georgia Institute of Technology.
- Yip, S. (2005) (ed.). Handbook of Materials Modelling. Volume I: methods and models, Springer, printed in the Netherlands. 1-14.