

T8 - Phase Analysis

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ARCHAEOLOGICAL STUDY OF DUTCH TIN SPOONS FROM AMSTERDAM: 1350 -1750 AD A NEUTRON SCATTERING STUDY**D.Visser^a, W. Kockelmann^b, P. Hallebeek^c, J. Veerkamp^d, W. Krook^d**^a NWO-Physics, ISIS Facility, Rutherford Appleton Laboratory, Chilton Didcot OX11 0QX, UK,^b ISIS Facility, Rutherford Appleton Laboratory, Chilton Didcot OX110QX, UK^c Netherlands Institute for Cultural Heritage (ICN), Department of Conservation Science, Gabriël Metsustraat 8, 1071 EA Amsterdam, The Netherlands^d Archaeology Amsterdam, Noordermarkt 45, 1015 NA Amsterdam, The Netherlands

The Department of Archaeology from Amsterdam excavated over time more than 600 complete tin spoons and numerous incomplete parts within its town boundaries. The earliest types of these spoons date back to ca 1350 AD, while the latest are from ca 1750 AD. A large number of these spoons were investigated by X-ray fluorescence spectroscopy in order to determine their metal content. For this purpose the patina had to be removed over an area of 5 x 5 mm. The patina itself was investigated by X-ray diffraction. The objects were archaeologically dated, facilitated by hammer or rose marks on some of them. A large variation in Pb content has been found in these objects. Early spoons (1350-1450) may contain up to 40-50% Pb. Due to the health hazards of Pb, the production of tin spoons was regulated and overseen by the Guild after 1530. Only a low Pb content (~5-10 %) was allowed from then on. The X-ray data seem to suggest that this control was more or less lost after 1600.

In this paper we present first results on a time-of-flight neutron diffraction study on spoon fragments and complete

tin spoons from different time periods, carried out on the ROTAX neutron diffractometer of the ISIS Facility at the Rutherford Appleton Laboratory, UK. Neutron scattering permits to perform non-destructive quantitative analyses of the metal-alloy phases present in the bulk of the spoon parts (scoop, handle), and to look at the microstructure of each of the constituent phases, that include Sn, Pb and Sn-Pb alloys as well as SnO. In addition, texture analyses on some of the objects were carried out in order to obtain information on the orientation distribution of grains and, hence, to shed light on the manufacturing methods.

The diffraction patterns of 20 dated spoons over the period 1400 - 1750 AD were obtained. Large variations in Pb content were found for different objects whilst there is little variation of the metal composition of scoop and handle of one and the same spoon. The surface patina is for most cases not visible in the neutron data.

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PHASE CONSTITUTION AND STRUCTURE RIETVELD REFINEMENT IN THE SYSTEM $\text{La}_{1-x}\text{Zr}_x\text{MnO}_{3+}$ ($0 < x < 0.3$)**E. Syskakis, A. Gantis and M. Calamitotu***University of Athens, Solid State Physics Dept., Panepistimiopolis Zografos 157 84, Athens, Greece*

LaMnO_{3+} manganites doped with tetravalent ions at the La site are possible candidates for n-type conductivity. They belong to the family of rotationally distorted mixed valence perovskites with the lattice distortions playing significant role in their physical properties [1]. We report here the synthesis and structural study of the nominally composed $\text{La}_{1-x}\text{Zr}_x\text{MnO}_{3+}$ ($0 < x < 0.30$). Polycrystalline samples have been prepared by the solid state reaction method with thermal treatments in air at 1200-1670K. The XRD Rietveld refinement analysis (Fig.1) showed that all samples contain the main orthorhombic (Pnma) phase $\text{La}_{1-z}\text{Zr}_z\text{MnO}_{3+}$, the cubic (Fd3m) pyrochlore $\text{La}_2\text{Zr}_2\text{O}_7$ as well as a cubically stabilized (Fm3m) ZrO_2 phase. The amount of the latter two phases, depending on the final treatment temperature, increases in general with increasing

Zr content. No manganese oxide phase could be detected. This indicates that the solubility of Zr is limited in the perovskite structure. Moreover it is found that monoclinic ZrO_2 can be stabilised to cubic (Fm3m) by the presence of La and Mn.

The lattice constants and cell volume behaviour on Zr content of the main phase,

$\text{La}_{1-z}\text{Zr}_z\text{MnO}_{3+}$, depends on the final treatment temperature (1570 and 1670K respectively). The refinement of the occupation of the La/Zr site could not give clear results about the actual substitution level of Zr, due to the overlapping of peaks in the multiphase system $\text{La}_{1-x}\text{Zr}_x\text{MnO}_{3+}$. However characteristic changes at the La site have been observed by the Bond Valence Sum (BVS) values at the La site which are indicative of the stress of the perovskite cell

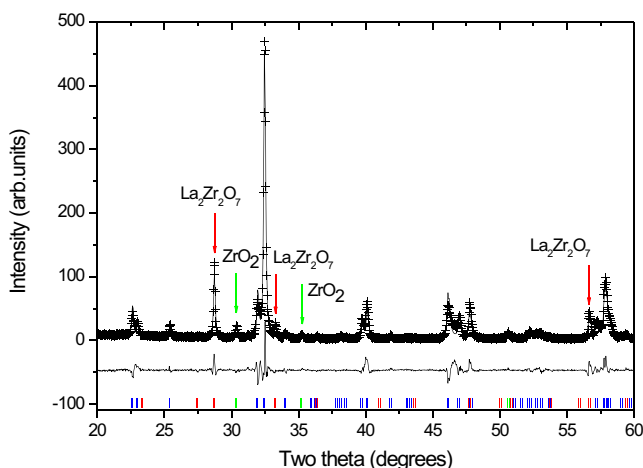


Figure 1. Experimental and calculated XRD pattern for sample with 30mole % Zr.

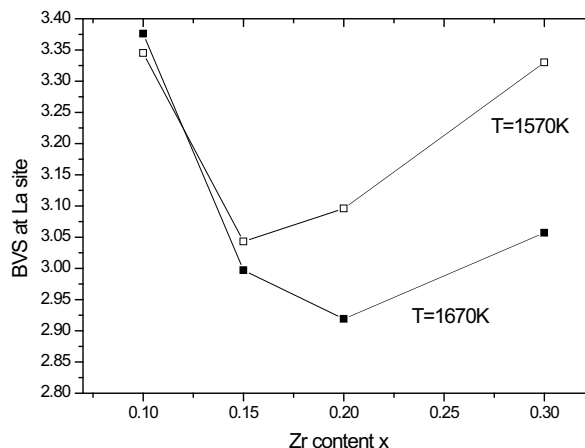


Figure 2. Bond Valence Sum values at the La site. Dotted line: the nominal value 3 for La

around the La atom. BVS values have been calculated according to $V_i \exp \frac{R_0 - R_i}{B}$ from the La-O refined in-

teratomic distances [2]. They exhibit a minimum, close to the nominal value 3 for an unstressed La^{+3} ion, at $x = 0.15$ ($x = 0.2$) for samples annealed at 1570 K (1670 K) (fig.2). This suggests that the stresses of the perovskite cell are relaxed at those nominal compositions by the substitution of the smaller Zr^{+4} for the bigger La^{+3} ion and/or by the La deficiency. The MnO_6 octahedron distortions show also a minimum at $x = 0.2$. Electrical conductivity and ac susceptibility data are consistent with the structural analysis showing limited solubility of Zr in the perovskite structure. They however stronger indicate that the incorporation of Zr into the orthorhombic

$\text{La}_{1-x}\text{Zr}_x\text{MnO}_{3+}$ structure is prohibiting the double exchange interaction mechanism.

Acknowledgment

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STRUCTURAL STUDY OF ACTIVE PHARMACEUTICAL INGREDIENT – TOLTERODINE TARTRATE

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Tolterodin tartrate ($\text{C}_{26}\text{H}_{37}\text{NO}_7$) is used as a medication, which allows the bladder to relax and hold more urine [1]. It is helpful for people with small or high-pressure bladders [2].

Following the new synthesis of tolterodin tartrate, series of crystallization experiments were made with a view to prepare polymorphs, solvates or hydrates. Crystalline material was studied using both x-ray powder and single crystal diffraction. Neither CSD nor PDF contain information about the crystal structure.

Crystals possess needle-like habit. Due to this fact, diffractograms of tolterodine tartrate show big preference orientation. We also present influence of grinding to the intensity and FWHM of the peaks.

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T8 - P144

CHARACTERIZATION OF POTENTIAL NON-EROSIVE ORAL DRUG DELIVERY SYSTEMS: MESOPOROUS MCM-41 AND SILICON POWDERS

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The adsorption of active pharmaceutical molecules into stable, non-erosive mesoporous materials offers potential to control (delay) drug release, enhance drug dissolution, promote drug permeation across the intestinal cell wall (bioavailability) and improve drug stability under the extreme environment of the gastro-intestinal tract when administered orally. Different types of drug molecules (including macromolecules) can be adsorbed into mesoporous microparticles by modifying the pore architecture and surface chemistry of the carrier material.

Porous silicon (PSi), produced by anodizing Si(100) wafers, has already been proven to be a potential candidate for biomaterial applications as its toxicity has been studied *in vivo* [1]. Synthetic MCM-41 type molecular sieves consist of a uniform mesopore channel structure providing a very narrow pore size distribution [2]. Some preliminary *in vitro* studies indicate it has very low cytotoxicity providing predictive evidence of its biocompatibility [3]. Recently, some applications have been reported regarding both PSi and MCM-41 as promising drug delivery vehicles [4, 5].

In the present study, drug loaded microparticles of calcined siliceous MCM-41 and thermally-carbonized porous silicon (TCPSi) were produced and characterized. First, the starting materials are examined to verify that their synthesis has been successful considering the structural properties and phase purities. Secondly, the drug loaded matrices are studied to determine if the loading procedure has been efficient i.e. to determine if the drug has adsorbed into the pores. In the case of successful loading, the structural state of the drug is qualified (crystalline or molecular amorphous) and the amount adsorbed quantified (w/w%). The characterization methods used for this are x-ray powder diffraction (XRPD), nitrogen adsorption (SSA and pore size distribution), pycnometry (density), differential scanning calorimetry (DSC) and thermogravimetry (TG). Ibuprofen, a well-known analgesic was selected as the model drug. Ibuprofen was loaded into sieved TCPSi and MCM-41 microparticles (<38 μm) using saturated ethanol (EtOH, 99.5 %) solutions. After a loading period the samples were filtered and dried at suitable temperatures.

X-ray powder diffraction can be used to support the other methods in verifying of successful drug loading and to determine the crystalline state of the adsorbed drug. The XRPD patterns of the pure and loaded matrices are presented in Figure 1. In the case of TCPSi, peaks characteristic for ibuprofen are obtained from the loaded microparticles in addition to the intense (111) reflection of silicon. This indicates that ibuprofen has crystallized in the

pores or onto the particle surfaces (or both). This can be validated using DSC and the results showed a significant amount of crystallized ibuprofen in the pores with only a fraction on the particle surfaces.

The XRPD patterns of the un-/loaded MCM-41 show four characteristic reflections of the uniform mesopore channel structure with (100) and (210) indicated in addition to the hump of the amorphous silicate pore walls between 15-35 (2θ). No ibuprofen reflections are observed in the pattern of the loaded MCM-41 sample. The pore centre repeat distance (a₀) of the hexagonal pore structure of MCM-41 correlates with the d₁₀₀ interplanar spacing value and can be calculated using the simple equation a₀=2d₁₀₀/√3 [2]. No major change in the pore repeat distance is detected suggesting maintained stability of the MCM-41 structure. The drug loading clearly decreases the intensities of the reflections with the (210) peak nearly disappearing. This is caused by the increase of the scattering power of the filling pore thus decreasing the scattering contrast between the pores and the walls [6]. These results indicate adsorbed substance in amorphous molecular state in the pores of the MCM-41 matrix.

The effect of the materials different pore sizes on the drug loading and crystallinity will be discussed in the poster. In addition the results for the amount of ibuprofen loaded into TCPSi and MCM-41 will be given. The combi-

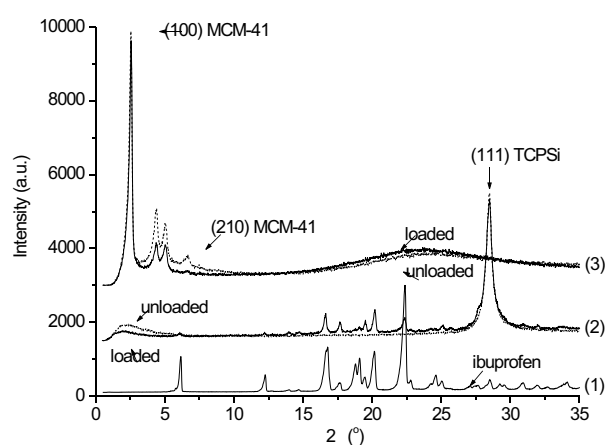


Figure 1. XRPD patterns of (1) ibuprofen [intensity scaled 1:10], (2) un-/loaded TCPSi and (3) un-/loaded MCM-41.

nation of XRPD and the other methods employed here



yield a good insight into the drug loading properties of the studied materials.

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T8 - P145

STRUCTURAL ASPECTS OF CATHODE MATERIALS IN RECHARGEABLE LITHIUM-ION BATTERIES

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The road to today's lithium-ion battery has passed through a number of structurally interesting steps. The first concept was the *lithium-polymer battery*; typically: $\langle \text{Li} \mid \text{polymer electrolyte} \mid \text{V}_6\text{O}_{13} \rangle$, where lithium inserts into the V_6O_{13} structure under the formation of a series of 5-6 different phases [1].

However, safety problems with using metallic lithium provoked the development of the "rocking-chair" *lithium-ion battery*; typically: $\langle \text{graphite} \mid \text{polymer or liquid electrolyte} \mid \text{LiMO}_x \rangle$, where M is Mn or Co [2]. More recently, LiFePO_4 (an olivine-type mineral) has been found to be a promising cathode material. It is an environmentally friendly material with several positive features: high capacity (170 mAh/g), light weight and low cost. Its main disadvantage is its poor electronic conductivity, resulting in limited utilisation of the material [3]. Three main strategies have been used to overcome this problem:

Coating with some high conductivity material, e.g. carbon.

Reducing particle-size to give better utilisation.

Substitution of other metal ions into the Fe-site.

We are currently working with all three approaches; we present here three examples where structural studies have provided valuable information:

LiFePO_4 has been synthesised in an aqueous solution using wet-chemistry techniques, to bring the particle sizes down to nano dimensions. Powder XRD has confirmed that the structure of this nano-material is indeed that of LiFePO_4 .

Partial substitution of the Fe-site in LiFePO_4 by some other transition metal (Mn and Co) has been probed with neutron powder diffraction; this is especially useful because of the exceptional ND contrast between Fe and the Mn or Co nuclei.

In-situ XRD to probe the charge and discharge reactions by monitoring the phases of LiFePO_4 versus FePO_4 .

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T8 - P146**NEUTRON AND X-RAY RIETVELD QUANTITATIVE PHASE ANALYSIS OF INDUSTRIAL PORTLAND CEMENT CLINKERS****O. Pritula¹, L. Smrčok¹, D. M. Többens², V. Langer³**¹*Institute of Inorganic Chemistry, Slovak Academy of Sciences, Dúbravská cesta 9, SK-845 36 Bratislava, Slovak Republic*²*BENSC, Hahn-Meitner Institute, Glienickerstrasse 100, D-14109 Berlin, Germany*³*Department of Environmental Inorganic Chemistry, Chalmers University of Technology, S-412 96 Gothenburg, Sweden*

Weight fractions of four dominant phases (C_3S , C_2S , C_4AF and C_3A) present in five industrial clinkers were estimated by a series of neutron and X-ray Rietveld refinements. Calculated powder patterns were derived from the structural data for triclinic and monoclinic C_3S , monoclinic C_2S , orthorhombic

C_4AF and cubic C_3A . Neutron diffraction data were collected with the high resolution E9 diffractometer (BENSC) using the wavelengths of 1.797 Å and 2.816 Å, X-ray diffraction data with a high resolution transmission diffractometer using $CoK_{\alpha 1}$ radiation. Elemental composition of the samples obtained by ESEM/EDX technique were in a good agreement with the data delivered by the producers. Convergence of the refinements was remarkably different for X-ray and for neutron data. Several re-

finements were not completed due to numerical instabilities. Neutron refinements were found to be more stable than X-ray, but there was not any notable difference in the final estimated phases' compositions. Calculated absolute deviates of phases' weight fractions were mostly within $\pm 10\%$, which for the less abundant phases corresponded to relative deviations within $\pm 50\%$.

Acknowledgement

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T8 - P147**APPLICATION OF X-RAY DIFFRACTION IN FORENSIC SCIENCE****M. Kotrlý***Institute of Criminalistics Prague, Strojnicka 27, P.Box 62/KUP, 170 82 Prague 7, Czech Republic*

Use of XRD analysis does not belong among traditional methods of forensic science. This situation arose due to more complex instrumentation in the past, and also some other effects. In the forensic field, the analyst often faces a problem of identifying completely unknown phases in a mixture, and real possibility of implementation into forensic science was allowed only by development of identification software and electronic databases, which enabled processing of similar tasks.

XRD analysis started to be employed at the Institute of Criminalistics Prague in the 60s of the past century for identifying short-circuit products on copper conductors. In the 90s a lab with two goniometers in classic Bragg-Brentan reflex geometry and with other radiation source, which is utilized for Guinier and Gandolfi camera with classic film. Film carriers from these chambers are subsequently digitalized and evaluated as records from both goniometers. ZDS software, ICDD database and client databases are used for evaluation.

Importance of XRD and phase analysis in forensic science lies namely in:

- the possibility of analysis of relatively small-volume samples

- the method is relatively non-destructive (the sample can be used for further analyses even after possible powdering) – it follows from the mentioned fact that probative value of the material is preserved
- the method allows exact identification of phases in a mixture (unlike other instrumental chemical methods)
- in majority of cases it is possible to determine quantity (or semiquantity) of substance in a mixture
- the method is conclusive for potential court proceedings

Currently, XRD analysis is employed in 7 main areas:
1) Complex analyses and soil comparisons – the method is indispensable for determination of clay fraction. Other available analytical methods (EDS/WDS, XRF, FTIR, etc.) are not able of performing exact determination of phases, namely aluminosilicates. Own calibration sets are used for semiquantitative XRD analysis.
2) Determination of explosives and post-blast residues – direct determination of inorganic and organic phases of explosives and post-blast residues (following the sampling of



the post-blast scene and possible separations and concentrations of the material). XRD methods are used also for evaluation of the effect of separation and concentration methods of post-blast residues during their testing. (The post-blast scene is usually very difficult to analyse, since there are particles of all kinds of materials destroyed by the explosion, in which there are relics of post-blast residues dispersed all over, often below the detection limits. That is why different concentration and separation procedures able of reducing the content of contaminants. During tests of effect of these methods, phase XRD analysis with semi-quantitative evaluation proved the most convenient.

3) Pigments and paints analyses – XRD methods are used in complex expert examination during determination of phases of artworks (paintings, sculptures etc.), car paints (analyses of abrasions and fragments after traffic accidents), analysis and comparison of lacquer systems of tools and instruments, lacquer systems used in building industry and some analyses of printing colours and paints.

Examination of pigments of artworks is requested for solving the following issues:

- partial examination to determine the age of the painting (on the basis of used pigments, canvass type etc.)
- confirmation of originality of adjustments of the paintings, or affiliation of the painting and the frame (e.g. confirmation that the pictures were in original frames prior to the theft)
- distinguishing the original from forgery (the most complex expert examinations, in which an expert from the National Gallery participates)

4) Identification of type and origin of goods – customs and financial frauds, counterfeiting of original branded products, money laundering. XRD methods belong to a complex of methods used for determination of the exact type of goods or material (e.g. materials for semiconductor compo-

ments, fertilizers and pesticides, special heat-resistant materials, precious stones, etc.)

5) Identification of unknown substances, potential poisons and contaminants - these analyses are requested for complex analyses of matters that can harm the environment or are subject to special treatment (industrial accidents and leakage of dangerous substances, attempts to dispose of these substances on ordinary waste dumps, threatening and extortionate letters – currently frequent packets with unknown white powder etc.). For these types of analyses it is necessary to use special user databases comprising standards of poisons and substances, which are not included in common commercial databases.

6) Confirmation of degraded skeletal discovery – burnt, fragments damaged or unusual to such extent that anthropologists require confirmation of composition. In this sphere, confirmation of phases, which are present in bone construction.

7) Quantitative drug analyses – with the aid of powder diffraction it is possible to reach higher precision of quantitative analysis of some narcotic mixtures than with instrumental methods of organic analysis (FTIR, GC). XRD analysis is in many cases faster and sample preparation is simpler.

Further development of XRD methods in forensic science will be perhaps brought by capillary optics and its application in micro diffraction, which even today allows execution of non-destructive analysis from points sized under 100 micrometers.

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T8 - P148

MICROSTRUCTURE AND PHASE COMPOSITION OF RAPIDLY SOLIDIFIED Al-Cr-Fe-Ti-Si ALLOY

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Rapidly solidified Al-6Cr-2Fe-1.5Ti-1Si (in wt.%) powder alloy was prepared by pressure (600 kPa) nitrogen atomization of melt. Microstructure and phase composition of various granulometric fractions were studied by using optical microscopy, SEM, TEM and XRD. It was found that the RS state is characterized by presence of various intermetallic phases in form of spheroids and irregular particles. The fraction of spheroids was found to increase, as

the powder particle size reduced, i.e. as the cooling rate increased. XRD and TEM investigations revealed the presence of quasi-crystalline icosahedral Al_{84,6}Cr_{15,4}, Al₈₂Fe₁₈, Al₅Fe₄Cr, Al₇₄Cr₂₀Si₆ and crystalline Al, Al₁₃Cr₂, Al₁₃Fe₄, Al₃FeSi phases. The coarser was the powder particle size, the lower was the fraction of quasi-crystalline phases. Upon annealing at 350°C, the quasi-crystalline phases decomposed to form Al₁₃Cr₂, Al₃FeSi and TiSi phases.

T8 -P149

STRUCTURAL ANALYSIS OF AUSTENITE NITRIDES**Antonín Buchal***VUT v Brně, FSI, ÚMI-OSFA Technická 2 616 69 Brno, Czech Republic*

Austenitic steel ion-nitriding exhibits very interesting conduct: there are no any nitrides, such Fe_3N , Fe_4N or Cr_3N , Cr_2N ...

The result of nitridation process is so called γ' -martensite (hexagonal structure with lattice parameters $a_0 = 0.2685$ nm $c_0 = 0.4752$ nm, sg. P $6_3/mmc$) and the regular γ -martensite ($a_0 = 0.2891$ nm sg. $Im3m$).

If a small amount of γ' - Fe_4N occurs, it is sputtered from surrounding atmosphere in ionization chamber. In any case it is not the product of reaction in solid state. If a diffractions of austenite occurs in diffraction record, it is from the layer not by nitrogen affected.

T8 - P150

X-RAY DIFFRACTION AND SEM STUDIES ON THE EFFECT OF TEMPERATURE ON THE FORMATION OF MAIN PHASE $\text{Sr}_2\text{MgSi}_2\text{O}_7$ USING A WET AND DRY METHOD FOR ITS PREPARATION**Ali Asghar Sabbagh Alvani¹, Fathollah Moztarzadeh², Ali Asghar Sarabi¹**¹*Department of Polymer Engineering, Amirkabir University of Technology, Tehran-Iran, P.o.Box 15875-4413*²*Faculty of Biomedical Eng. Amirkabir University of Technology, 424, Hafez Ave. Tehran Iran*

A pure Silicate host namely $\text{Sr}_2\text{MgSi}_2\text{O}_7$ as a support for a long-lasting afterglow when doped with rare-earth elements was sought after. To this end the process of obtaining firing temperatures of the above mentioned phase in normal condition was carried out using suggested wet and dry methods.

The degree of purity obtained in these preparatory methods as well as the different firing temperatures were studied by the aid of SEM and XRD techniques. It was

found that for the wet method there exists a temperature beyond which the amount of impurity remained constant. There also existed a temperature at which both the wet and dry methods gave the same amount of impurity. Beyond this temperature the wet method would be preferred.