Optical and Mechnical Studies of Potassium Nitrate (KNO_3) single crystal

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Abstract : A nonlinear optical crystal – Potassium Nitrate KNO3 was grown by slow evaporation of solution growth method in different Molar concentrations and their optical and mechanical properties were studied. The optical properties were studied by means of transmission and reflectance in the wavelength region 200–1200 nm. The optical transparency and lower cut off value of UV transmission were ascertained by recorded UV-Visible spectrum. Optical band gap of the crystals were found to be more than 3.5 eV. Functional groups of the grown crystals were identified through FTIR spectroscopy. Mechanical property of the grown KNO3 crystals was studied by Vickers micro hardness tester by varying the mass of the load.

IndexTerms **- Potassium nitrate, UV – Vis NIR, Optical band gap, FTIR Analysis, Microhardness**

INTRODUCTION

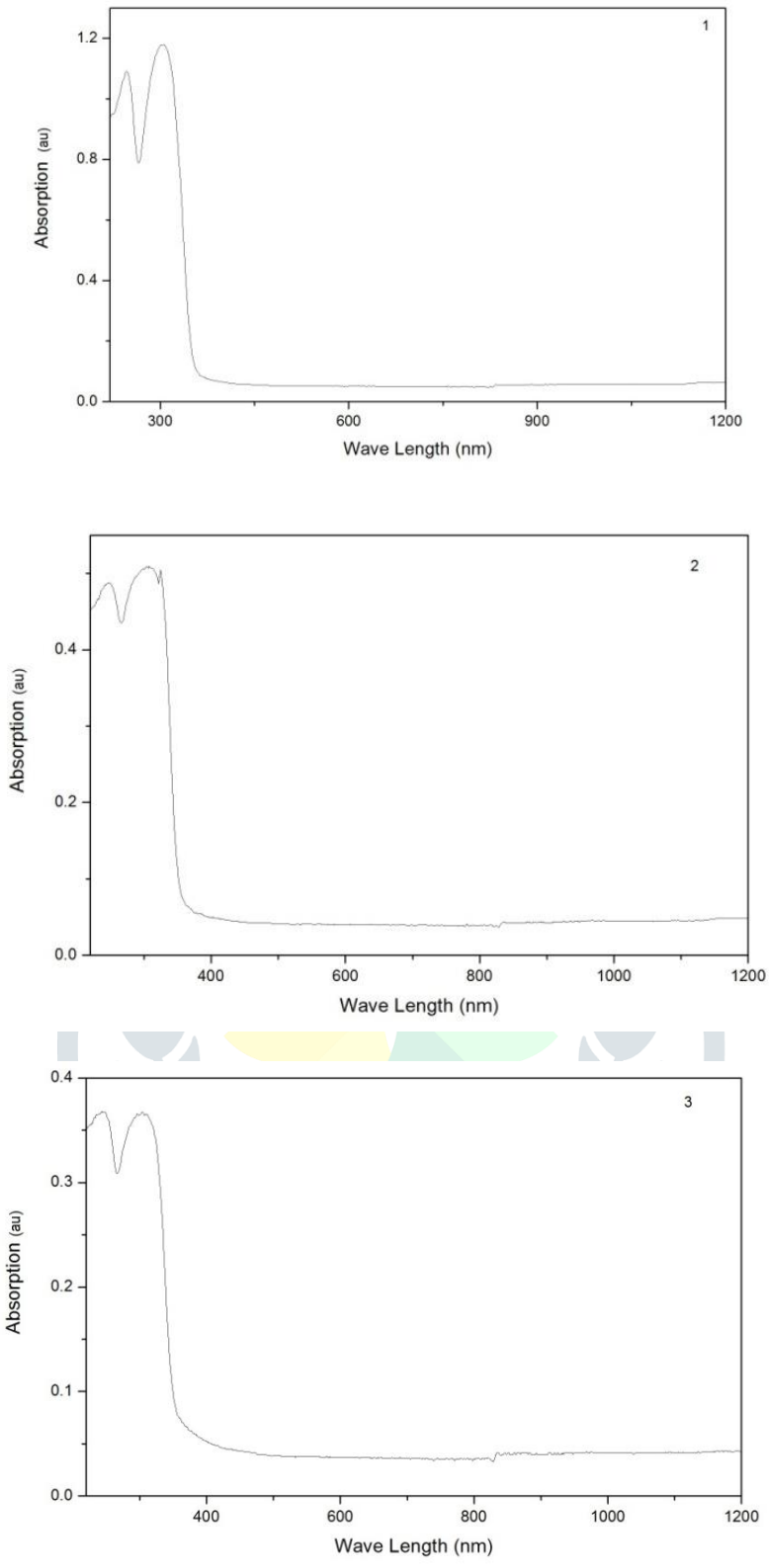
Potassium nitrate (KNO3), also known as saltpeter or niter, is a chemical compound consisting of potassium, nitrogen, and oxygen. Potassium nitrate (KNO3) is used as a fertilizer, food preservative and heat transfer agent in chemical industries. KNO3 is also essential in the production of xplosives, glass and steel [1, 2], Stump remover, thermal storage medium in power generation systems. The only known ore of KNO3 is caliche mined in Northern Chile [3]. Caliche is used in construction worldwide. The growth of KNO3 crystals has been examined by several authors [4]. KNO3 has several different phases depending upon the temperatures. When KNO3 is heated from room temperature to the transition point 129 OC, KNO3 transforms from phase II (orthorhombic) to phase I (trigonal) [5]. On cooling in the temperature range from 129 to 100 O C the phase I, KNO3 passes through another trigonal phase as phase III.

Synthesis and crystal growth:
Single crystals of pure Potassium nitrate (KNO3) were grown from aqueous solution by slow evaporation technique in room temperature. Analytic grade Potassium nitrate (HPLC-99.5%) of 3 gms, 4 gms and 5 gms were dissolved
in de-ionized water to get a saturated solution. Magnetic stirring for an hour was used to obtain a homogeneous solution. The solution was filtered and covered with a porous cover and kept in a dust free environment. Good quality single crystals of 0.6M, 0.8M, and 1M Potassium nitrate were harvested after four weeks. The grown crystals were subjected to characterization studies of UV-Vis, FTIR and microhardness.

Characterization:

UV-Visible Spectrum Analysis

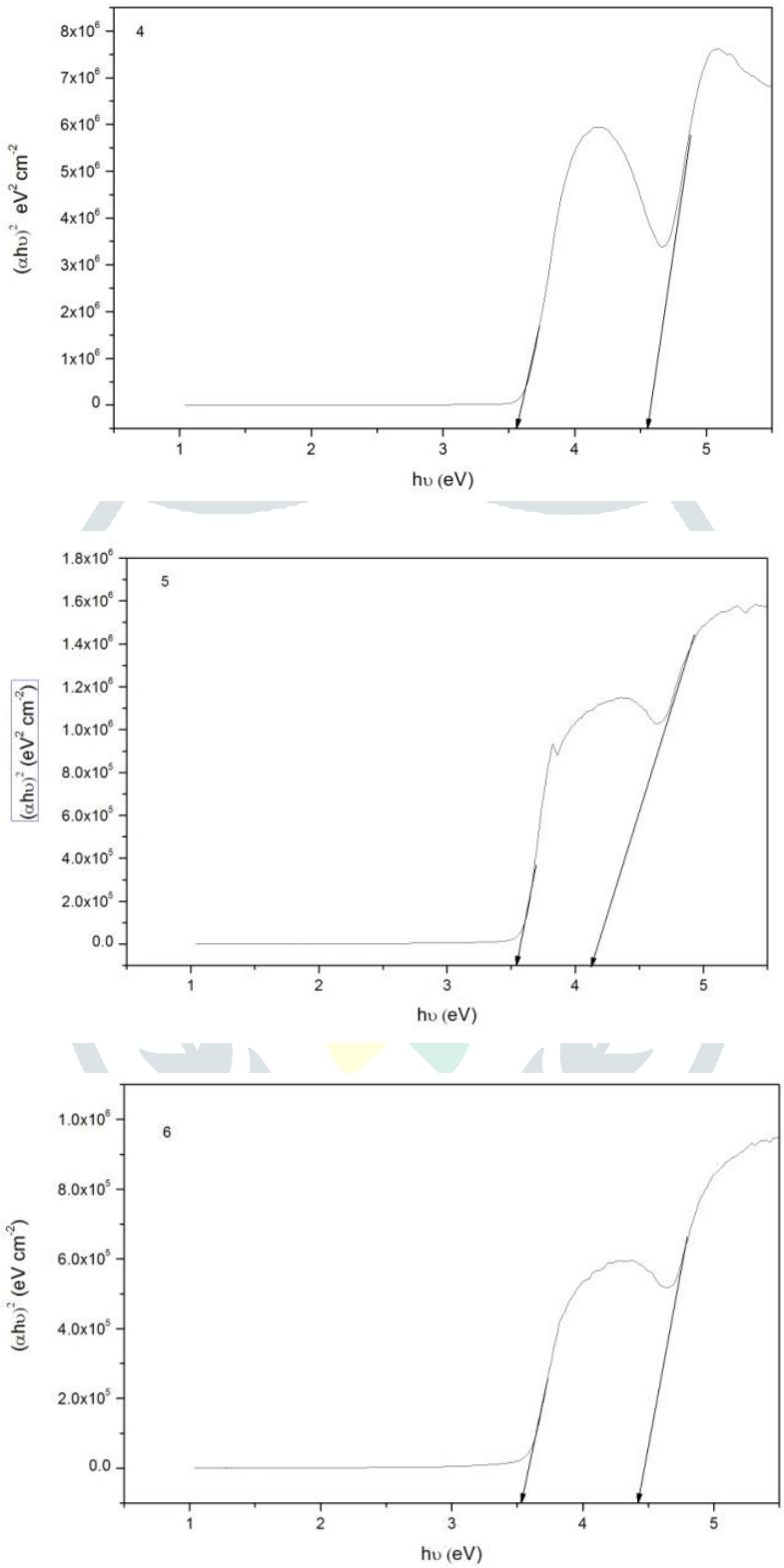
The transparent behavior of pure KNO3 crystals of value 0.6M, 0.8M and 1M in the entire UV–Visible region is clearly illustrated by its UV–Visible spectrum shown in Figures 1–3. The optical transmission spectrum of the grown KNO3 crystals was recorded in the wavelength range 200–1200 nm using a Shimadzu-UV2600 (Japan) UV-Vis spectrometer in Centre for Scientific & Applied Research (C-SAR), PSN CET. The optical absorption spectra of alkaline nitrate crystals consist of two bands [6-7]. From the UV-Visible absorption spectrum of pure KNO3 crystal the cut of wavelengths (λ) found to be 247 nm and 306 nm.
The UV- Visible spectrum of KNO3 shows that the short-wavelength (λ) absorption band corresponding to π-π* transition of electron and the long wave length (λ) absorption band corresponding to n-π* transition of electron. The pure KNO3 crystals show good transparency in the entire UV-Visible spectrum.



Figures 1–3: UV-Absorption spectrum of KNO3 crystals of 0.6M, 0.8M and 1M

2 OPTICAL BAND GAP

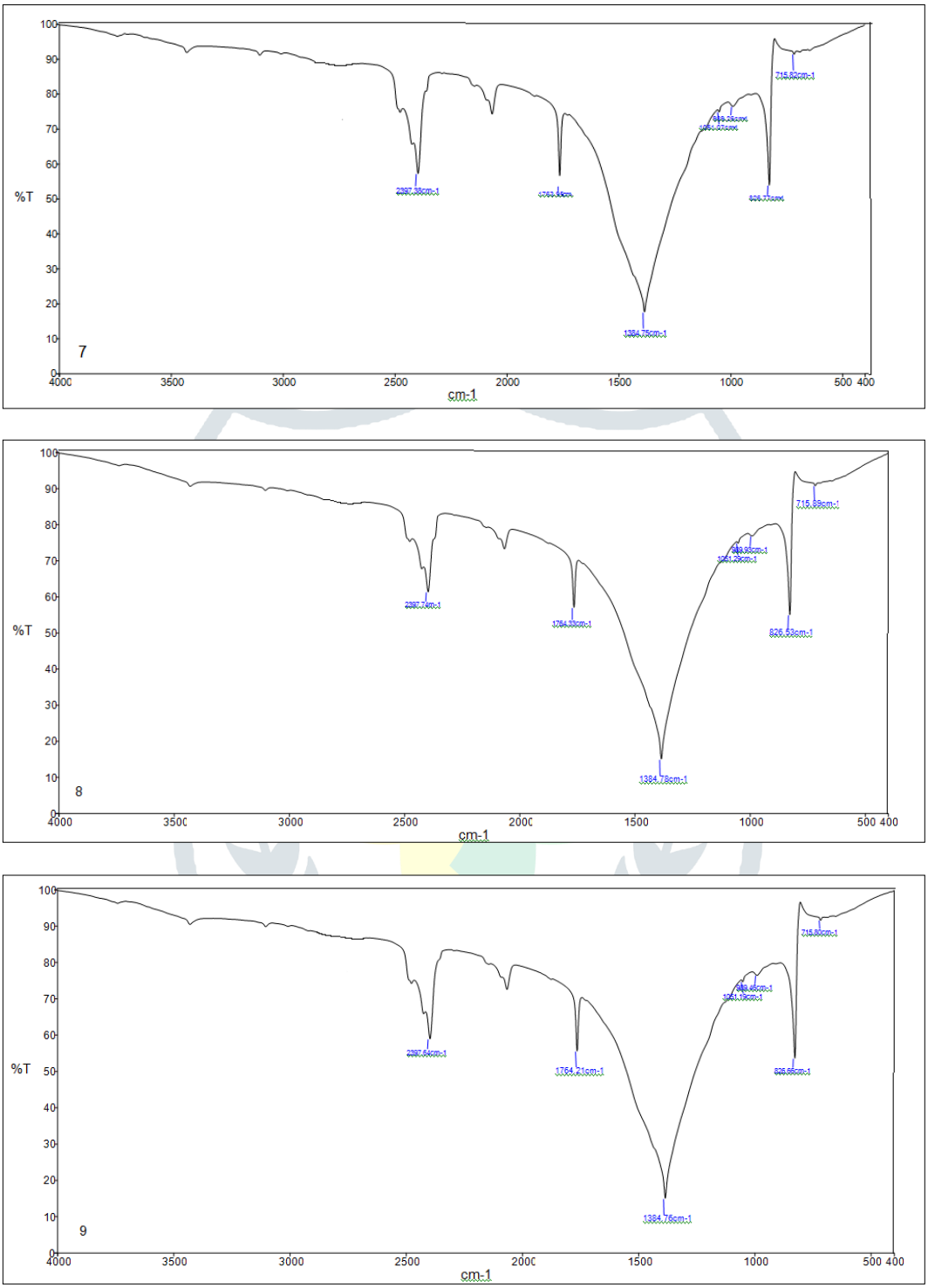
The dependence of optical absorption coefficient with the photon energy is useful to study the band structure and the type of transition of electrons. The optical band gap of the KNO3 samples determined from the Tauc's plot [8]
of $(\alpha h\nu)2$ as a function of photo energy ($h\nu$) as show in Figures (4-6). Because of the two electron transition π-π^*, n-π^* [8-10] two energy band gap values occurred. The optical band gap energy diagram shows two band gap energies of 3.5 & 4.6 eV, 3.5& 4.3 eV, 3.5 & 4.4 eV for the higher and lower photon energies of 0.6M, 0.8M and 1M respectively.



Figures 4–6: Tauc's plot for KNO3 crystals

3. FOURIER TRANSFORM INFRARED [FTIR] SPECTROSCOPY

Fourier Transform infrared [FTIR] Spectroscopy involves studying the stretching, bending twisting and vibrational modes of atoms in a molecule and used to identify the functional groups present in the compounds. KNO3 crystals were subjected and recorded by using Model Spectrum RX I spectrometer of Perkin Elmer in the range 400–4000 cm–1. The FTIR spectra of pure 0.6M, 0.8M and 1M KNO3 is shown in Figures (7–9) band Assignments are given in Table 1.



Figures 7–9: FTIR spectrum of KNO3 crystals

In the FTIR spectrum there is a sharp band in the region 800–870 cm^{-1} due to bending vibration of N-O group. The band at 826 cm^{-1} assigned to out of plane bending of N-O. Also a band in the region of 710–725 cm^{-1} corresponds to bending vibrations N-O. The band at 715 cm^{-1} represents Anti symmetric bending of N-O group. In the region between 1010–1400 cm^{-1}shows stretching vibrations of the NO_3 group. The bands at 1051 cm^{-1} and 1384 cm^{-1} are assigned to symmetric and anti symmetric stretching vibrations of the NO_3 group respectively [9-11].

Table 1: FTIR data table of KNO_3 single crystals

Wave number (cm^{-1})	Band assignment
1051	Symmetric stretching

715	Anti symmetric bending
1384	Anti symmetric stretching
826	Out of plane bending

4. MICROHARDNESS

The microhardness measurement was carried out for pure KNO3 Crystals having different molar value using Shimadzu HMV- 2T Vickers microhardness tester. The load P was varied from 25 g to 100 g and the time of indentation was kept constant as 10 s for all trials. The diagonal lengths of indentation (d) were measured in mm for various applied load (P) in g. The Vickers hardness number Hv was calculated using the relation

$$\mathbf{Hv = 1.8544\ P/d^2\ (kg/mm^2)}$$

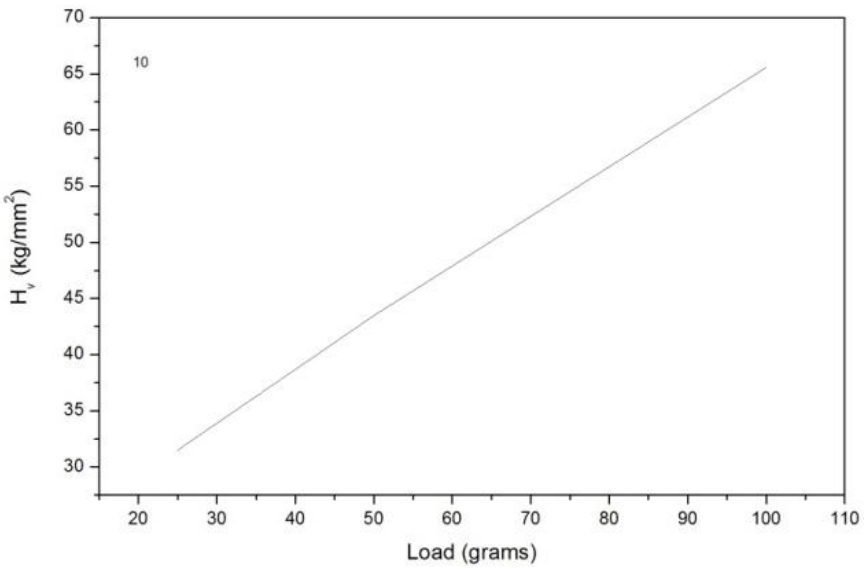
The variation of Hv with applied load P is shown in Figures (10–12). Figures show that Hv increases with an increase in the load. It reveals that hardness number increases with increasing applied load. This phenomenon is known as reverse indentation size effect (RISE).

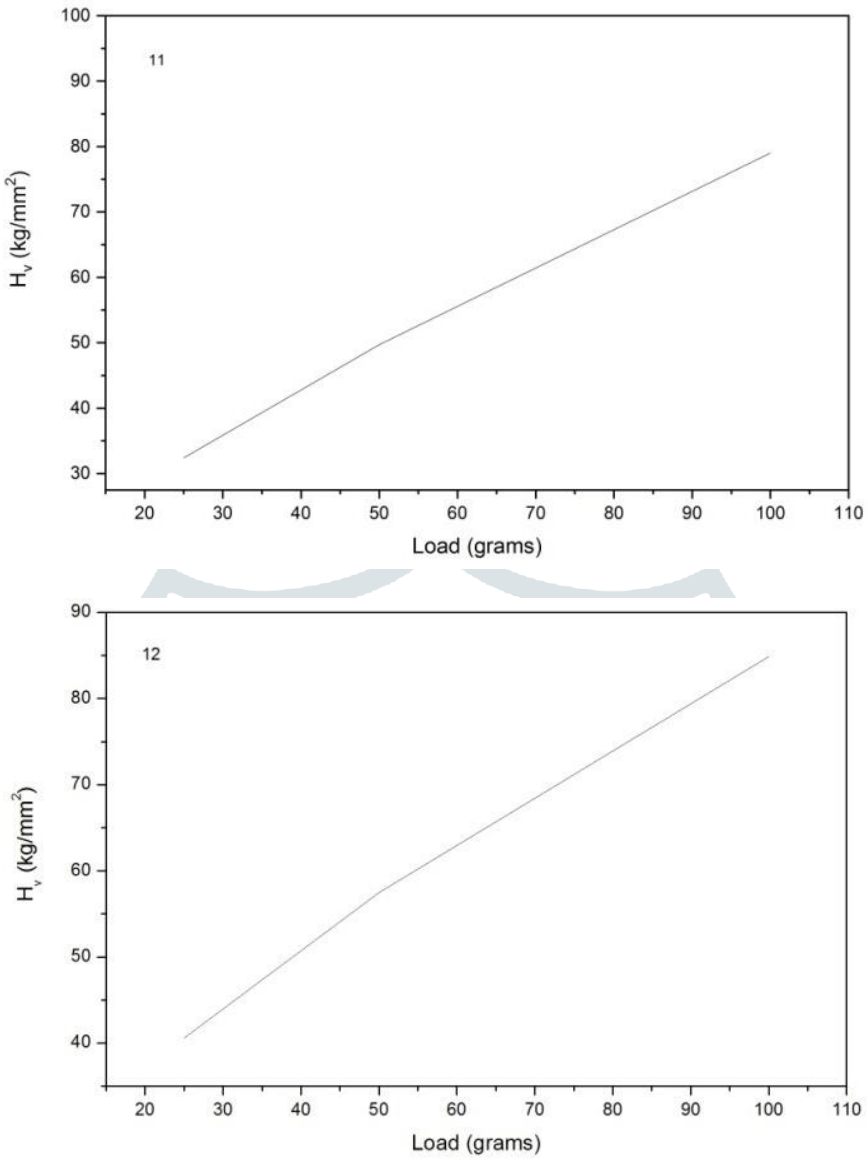
The Mayer's index number was calculated from the Mayer's law, which relates the load and indentation diagonal length.

$$\mathbf{P=k\ d^n}$$

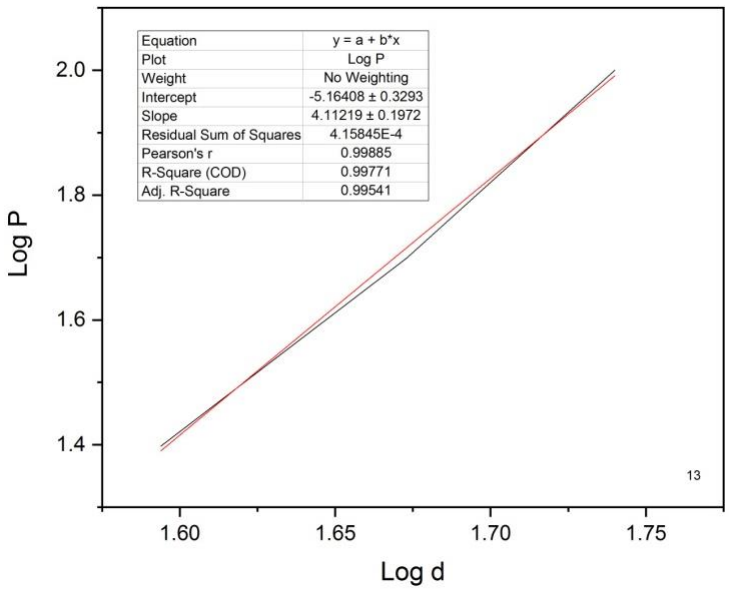
$$\mathbf{Log\ P=Log\ k\ +n\ Log\ d}$$

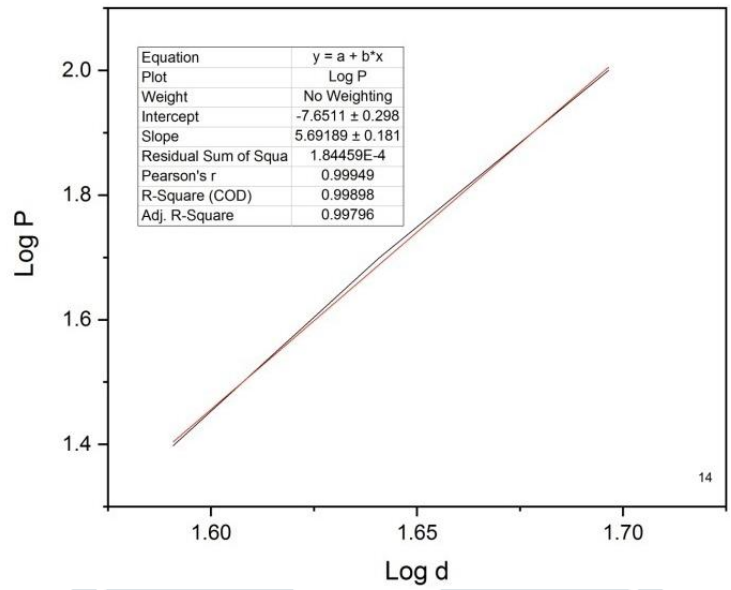
The Meyer's index (or work-hardening coefficient) n was calculated from the plot between log P versus log d as shown in Figures (13–15). According to Onitsch. E. M. [12] Manch and Hanneman [13] hard materials have n between 1 and 1.6; soft materials have n greater than 1.6. The value of The Meyer's index (n) was calculated using the least-square fitting method and it was found to be 4.11, 5.7, and 4.33 for pure KNO3 crystals. It is showing that KNO3 crystals belong to the soft material category.

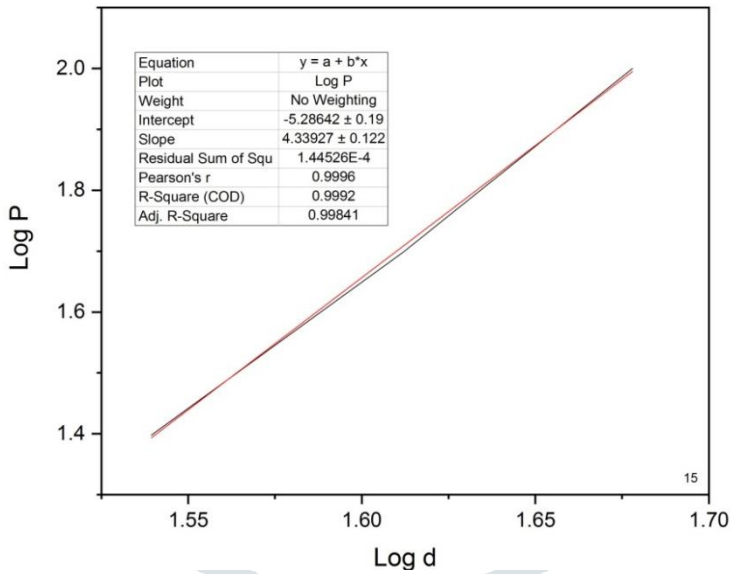




Figures 10–12: Variation of Vickers micro hardness number (Hv) with the applied load for Potassium nitrate crystals







Figures 13–15: Graph of log P versus log d for KNO3 crystals

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