Effect of Particle Size on the Physical and Mechanical Properties of Nano HA/HDPE Bio-Composite for Synthetic Bone Substitute

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ABSTRACT

The effect of using nano particle size of Hydroxyabatite to produce HA/HDPE bio-composite by Hot Pressing technique was studied by investigating the effect of particle size and production technique on the physical and mechanical properties of the bio-composite at different volume fraction of nano HA powder and different compression pressures.

Nano particle size and Hot Compression technique had great impact on the properties by reversing the behavior of the bio composite comparing with that using micro scale particle size in some of previous studies .The fracture strength and hardness increased with increasing the filler content by more than 200% for strength and 300-400% for micro hardness values , the densities increased with increasing filler content compressing pressure where as the porosity decreased.

The modification in mechanical properties due to filler particle size and production process enhanced the osteo- conductivity of biomaterial to use in different bone substitute applications.

Key words: Nano hydroxyabatite, Biomaterials, Mechanical Properties, Synthetic Bone Substitute.

تاثير الحجم الحبيبي على الخواص الفيزياوية والميكنيكية للمادة المتراكبة نانو هيدروكسي ابتايت/بولي اثلين

الخلاصة

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تم دراسة تأثيراستخدام حجم حبي بيذومدنانويل مسحوق الهايدروكسي ابتايت لإنتاج مادة مركبة بيولوجية باستخدام تقنية الكبس على الساخن وتأثير ذلك على الخواص الفيزياوية والميكانيكية للمادة المركبة عند نسب حجمية مختلفة من المادة المضافة النانوية وعند ضغوط كبس مختلفة. كان لاستخدام الحجم الحبيبي النانوي وتقنية الكبس على الساخن تأثر كبير جدا على الخواص بحيث عكس سلوك المادة المركبة مقارنة مع نفس المادة عند حجم حبيبي ذو مد مايكرو والتي تم تناولها في الدراسات السابقة.
لوحظ زيادة بمقاومة الكسرتصل الى نسبة 200% من مقاومة الكسر لنفس المادة بالمقاس المايكروي وزيادة في قيم الصلادة تصل الى 300-400% من قيم الصلادة لنفس المادة بالمقاس المايكروي. كما ان قيم الكثافة ازدادت مع زيادة محتوى المادة النانوية المضافة ومع زيادة ضغط الكبس كما قل تقيم المسامية. ان الزيادة الكبيرة في الخواص الميكانيكية وتحسن الخواص الفيزياوية للمادة المركبة جعل من الممكن استخدامها في مختلف تطبيقات الاحلال بدل مادة العظم الطبيعي.

INTRODUCTION

Currently, there is a significant need for improved synthetic materials, for use as orthopedic implants, to replace human bone lost or damaged due to disease (e.g. osteoporosis) and/or injury.

Certain ceramics, like calcium phosphates, have the special property of being bioactive, meaning that an interfacial bond between the implant and the surrounding bone forms, leading to good fixation, and generally no fibrous tissue encapsulation .

One such ceramic, hydroxyapatite (HA)($Ca_5(PO_4)_3OH$), the stable phase of calcium phosphate at body temperature and pH>4.2 , is one of the main constituents of natural bone (B70wt%) and is being investigated in a wide variety of forms for use in different bone implant applications [1,2,3,].

However, as HA is intrinsically poor in mechanical properties, bone replacement parts made from HA have been used only in non-loadbearing areas such as the ossicles in the middle ear Therefore, for full utilization of bioactive HA-based implants, improvements in mechanical properties are necessary.[4]

Hydroxyapatite (HA)-reinforced polymer biocomposites were first conceived by W. Bonfield and colleagues as a bone analog biomaterial enabling mechanical properties to be tailored to mimic those of bone tissue. Bone tissue exhibits a complex, hierarchical structure over several length scales beginning with a distinction between the more dense cortical bone in the diaphysis and less dense trabecular bone in the epiphyses of long bones, such as a human femur Figure (1). However, regardless of differences in intermediate levels of structure, the extracellular matrix (ECM) of all bone tissue is essentially constructed by mineralized collagen fibrils, which can be accurately represented as a two-phase composite comprising a collagen matrix reinforced with 40–50 vol.% (50–60 wt.%) apatite crystals Figure (1). The apatite crystals are nanoscale, plate-like,and elongated with a c-axis preferred orientation in directions of principal stress, such as the longitudinal anatomic axis of long bones.Thus, bone tissue exhibits anisotropic and inhomogeneous mechanical properties.[5]

Most of the research which investigated. The HA/HDPE bio-composite,was done in the micro scale of particle size and by using chemical process or Twin screw Hot Extrusion technique[6,7] ,but in this study we shall investigate the effect of nano size of HA on the mechanical and physical properties in order to create new nano composite material for bone repair and substitute by using Hot compression technique.

MATERIALS AND METHOD

2.1:Materials :

Materials used in this study were: 1.Hydroxyabatite99% pure with particle size of 20 nm with a nodular shape with and density of (3.140 gm/cm^3) supplied by M.K Nano (Canada , Toronto), the chemical analysis explained in table no.(1) .

2.HDPE with particle size of 5 um and density of (0.95 gm/cm^3) supplied by (China ,Shanghai) .

The properties of HDPE are listed in Table (2).

Composite Synthetic method

At first, powders were mixed as dry in a ball mill for 12 hr, then shaped by using Hot Die Compression Technique at 140 C$^\circ$,compression temperature and 56,85,112,140 MPa as compression Pressures ,for 0.5 hr as a hot compression time.

The samples were shaped as a disk with 15 mm diameter and 6 mm thickness.

Physical Properties Evaluation

The densities of the samples were calculated using Archimedes method as in the following equations :[8,9]

Bulk density = (WD/Wa-Wb) x D …(1)

Bulk volume =(WD/Wa-Wb)x D …(2)

Apparent solid volume = (WD-Wb/D) …(3)

Apparent solid density =(WD/WD-Wb)x D … (4)

Where:

Wa= weight of test piece soaked and suspended in air

Wb= weight of test piece soaked in water and suspended in distilled water

WD= weight of test piece

The sintered samples are soaked in distilled water for (1) hr before measurment.

Porosity evaluation

The True porosity was calculated using the following relation :[8,9]

True porosity%=[1-bulk density/true density]x100 …(5)

Mechanical Properties

Tensile strength

Tensile strength for disk shaped samples was measured using Diametrical Compression Test.

In a diameter-compression test a disk specimen is loaded in compression edgewise along a diameter as showing in Figure (2). The loading generates a biaxial stress state in the specimen with a compressive principal stress in the direction of loading and a transverse tensile stress. These stresses are nearly constant for a significant fraction of the test specimen near the center of the disk. Their magnitudes are given by the relationships:[10,11]

$\sigma = 2P/ \Pi Dt$ …(6)

Where: P= load (N)

D= diameter of specimen (mm)

t= thickness of specimen(mm)

VickersMicrohardness

Vickers micro-hardness was carried in a Timeinstrument(Digital Micro Vickers Hardness Tester TH714), read a direct measurement of hardness . Pyramid indenter is used with applied load of (50 gm).

Chemical analysis

Chemical analysis were carried out using X-ray spectrometery to find the composition of Hydroxyapatite nano powder as shown in Table (1).

Results and Discussion

Physical Properties

Figures(3and 7)show the effect of compression pressure on the Bulk density and apparent solid density respectively at different volume fraction of nano HA as a filler materials ,from the tow figures above it could be seen that the densities increase with increasing compression pressure and HA vol% ,duo to good filler-matrix interlocking by increasing compression pressure and nano HA volume fraction.

Figure(4) explains the effect of filler content and compression pressure of the true porosity , it is clear that the porosity decreased with increasing filler volume fraction and compression pressure duo to the good densification process during the liquid phase sintering .

Figures (5and6) show the effect of compression pressure on the Bulk volume and Apparent solid volume at different nano HA volume fraction content.

The volume decreased with increasing compression pressure due to shrinkage in samples because of sealing the pores.

The Bulk volume increased with increasing the nano HA volume fraction content because the nano particles tends to coat the polymer particles during mechanical mixing .

Mechanical Properties

Figures (8and 9) show the effect of compression pressure on the tensile strength, and hardness at different volume fraction of nano HA filler .

The tensile strength and hardness of the composites are found to be increased with increasing the amount of HAp contents.

The tensile strength increased by more than 200% comparing with the previous studies[12,13].

The Vickers microhardness found to increase by 300 to 400% comparing with previous studies[14].

From the results, it is clear that the mechanical properties have been improved significantly with high nanoparticle loading capability, which means that the composite tends to be used as a biomaterial with high osteoconductivity . The enhancement in tensile strength and hardness in case of HAp/HDPEnano composite could be attributed to the excellent bonding at the nano particle -polymer matrix interface.[15]

By comparing these results with previous studies which had investigated the mechanical properties of HA/HDPE composite with particle size with micro scale , a great difference in the behavior of HA as a filler is noticed ,for example the Tensile strength decreased with increasing HA content .

The reduction in strength with increasing the filler content in micro scale may be explained by the presence of weak bond at the particle-matrix interface ,this type of interface forms as a result of mechanical interlocking produced by the shrinkage of polymer matrix on to filler particles while cooling the samples from compounding temperatures[12].

CONCLUSIONS

The use of nano HA particle size affects the mechanical ,and physical properties in positive way which led to reverse the behavior of HA totally comparing with using micro scale particle size . Maximum tensile strength was achieved at 30 volume fraction of HA at 58 Mpa compression pressure ,while the maximum hardness was achieved at 40 volume fraction of HA at 140 Mpa compression pressure.

Optimum density was achieved at 40 volume fraction of HA at 112 Mpa compression pressure.

The nano particle size affects directly the mechanical and physical properties and produced biocomposite with suitable properties not for bone repair only ,but for bone substitution .

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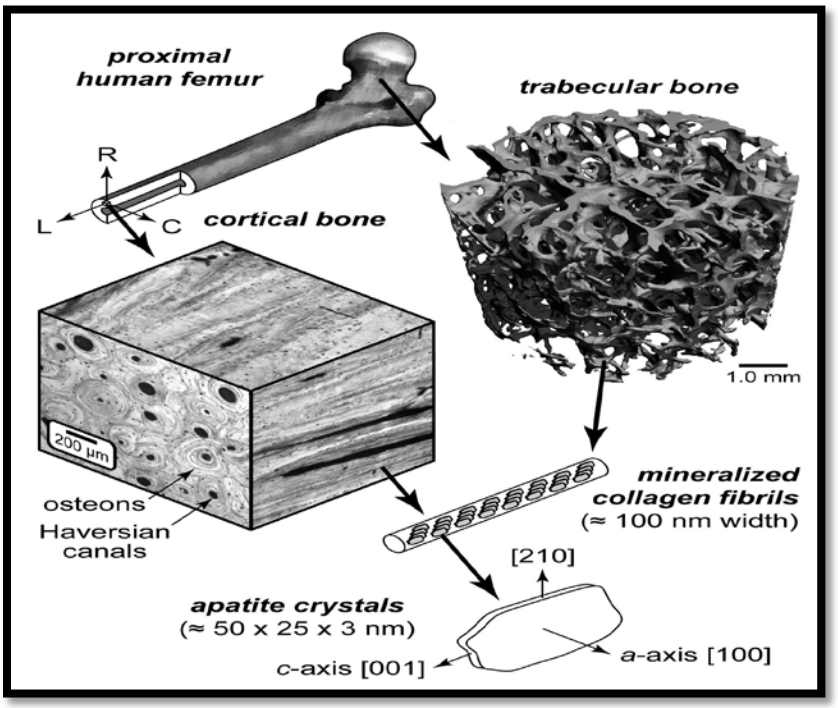


FIGURE(1): A schematic diagram showing selected levels of the hierarchical structure in human bone tissue. The microstructures of cortical and trabecular bone are shown using reflected light micrographs and a three-dimensional reconstruction from micro-computed tomography, respectively. Note that up to several levels of hierarchical structure, including further differences between cortical and trabecular bone, are not shown due to space constraints and in order to emphasize the commonality of mineralized collagen fibrils at the most fundamental level of structure. The longitudinal (L), radial (R), and circumferential (C) anatomic axes of the femur are shown relative to structural features.[2]

Table (1) Chemical analysis of nano HA powder.

HA	Sulfate	Chloride	Heavy metals	Loss on drying
99% pure	(0.048%max):0.025	(0.05%max):0.02	(10ppm max):7.0	(1.0% max):0.75

Table (2) Properties of HDPE.

HDPE cable material Property		Typical Value
Mass density g/cm 3		0.941-0.959
Melt flow Rate(190 centidegree /2.16kg)g/10min		≤1.0
Tensile yield strength MPa		≥19.0
Flexural modulus Mpa		≥965
Elongation@ break %		≥400
Impact Embrittlement temperature centidegree		MAX -118
Dielectric Constant	100KHZ	2.32
	1MHZ	2.31
Loss Tangent	100KHZ	MAX 0.0001
	1MHZ	MAX 0.0001
Volume resistivity OHM-CM		MIN 1x10 15

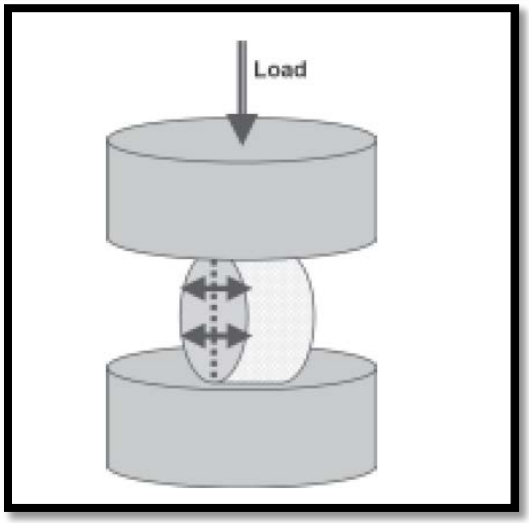


Figure (2) Schematic illustration of diametric compression test[8].

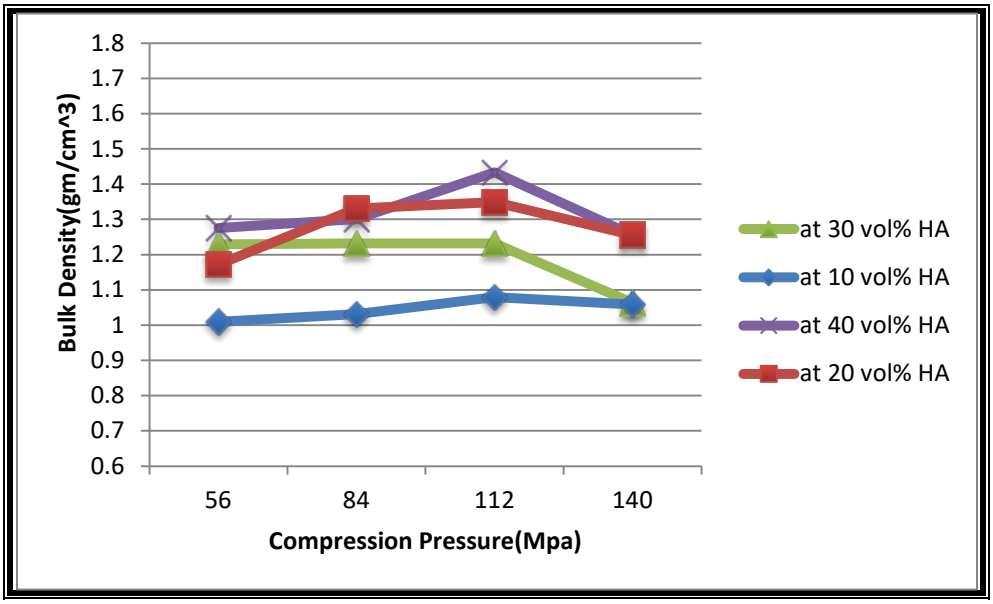
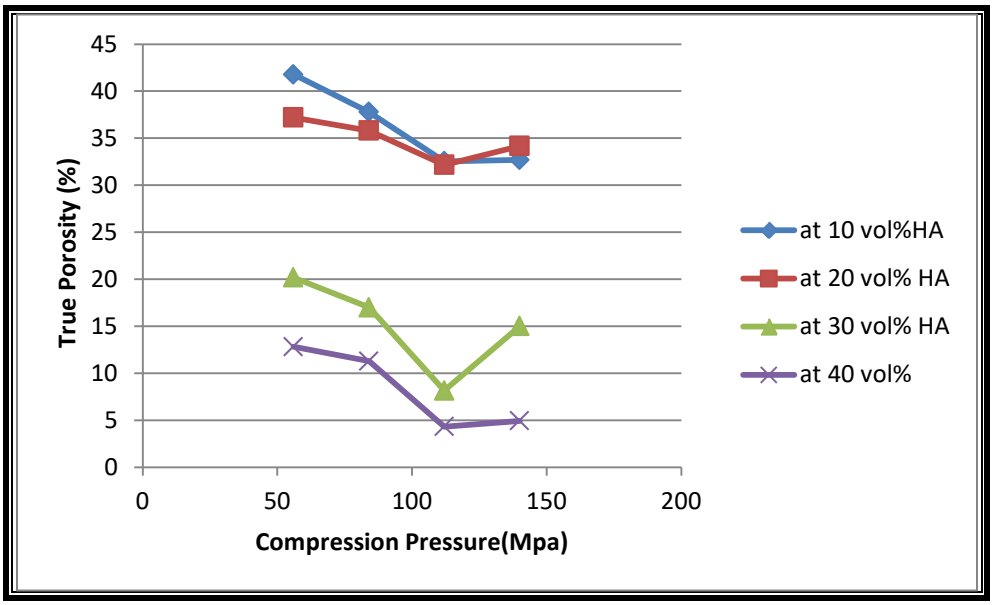
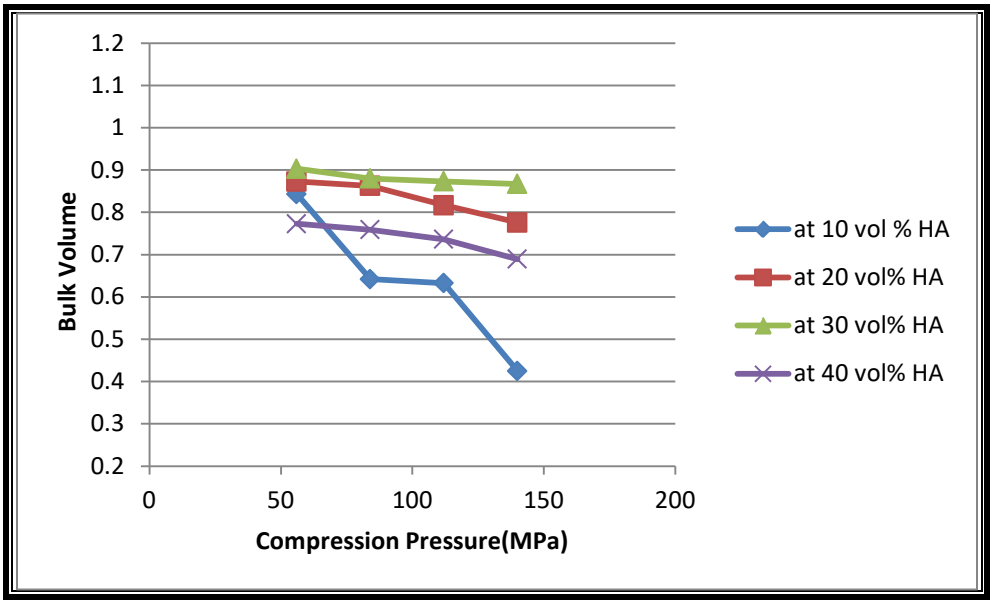


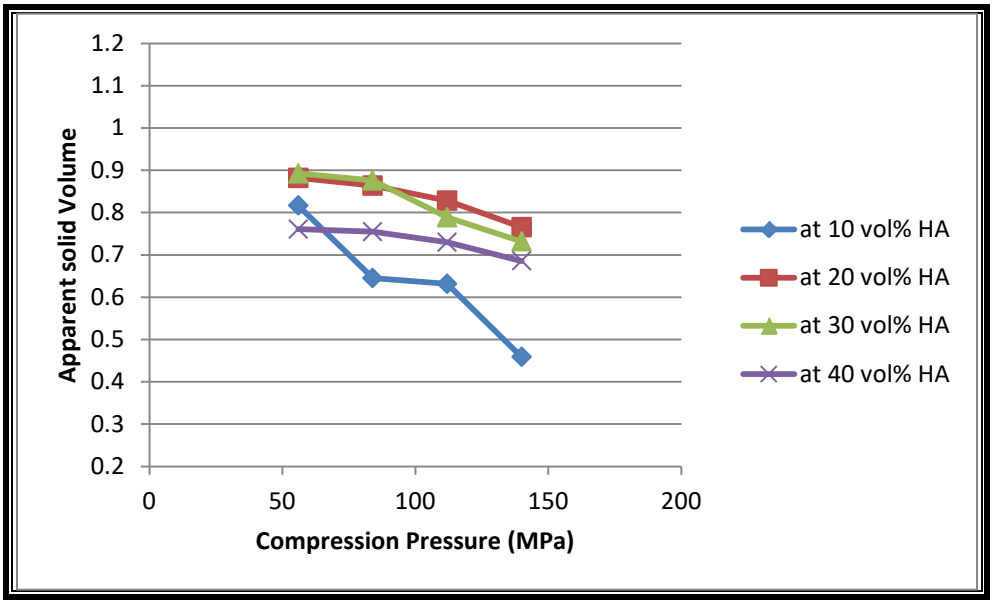
Figure (3)Effect of compression pressure on the Bulk Density at different vol% of HA.



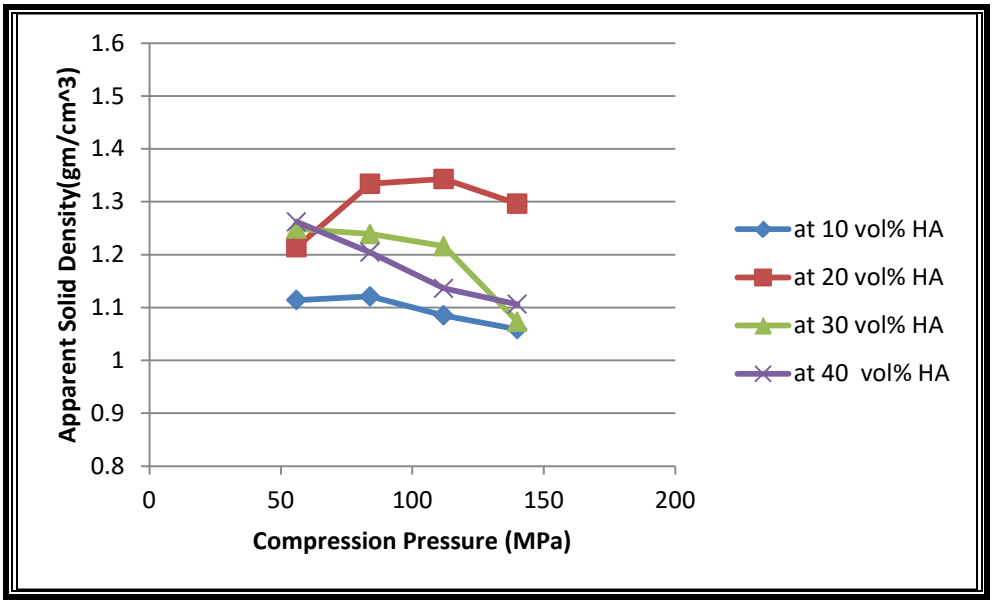
Figure(4):Effect of compression pressure on the True Porosity at different vol% of HA.



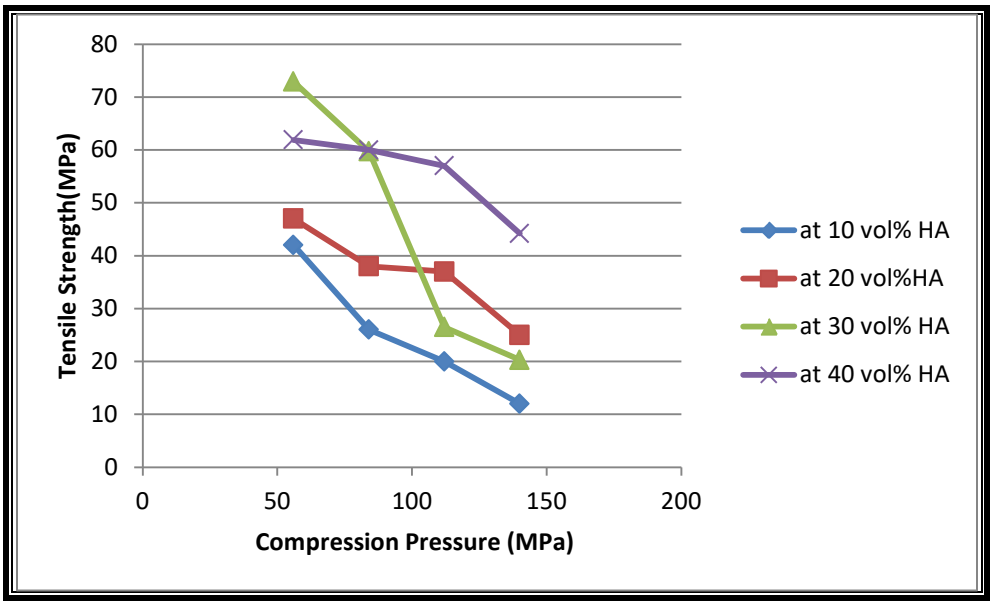
Figure(5) Effect of compression pressure on the Bulk Volume at different vol% of HA.



Figure(6) Effect of compression pressure on the Apparent Solid Volume at different vol% of HA.



Figure(7) Effect of compression pressure on the Apparent Solid Density at different vol% of HA.



Figure(8)Effect of compression pressure on the Tensile Strength at different vol% of HA

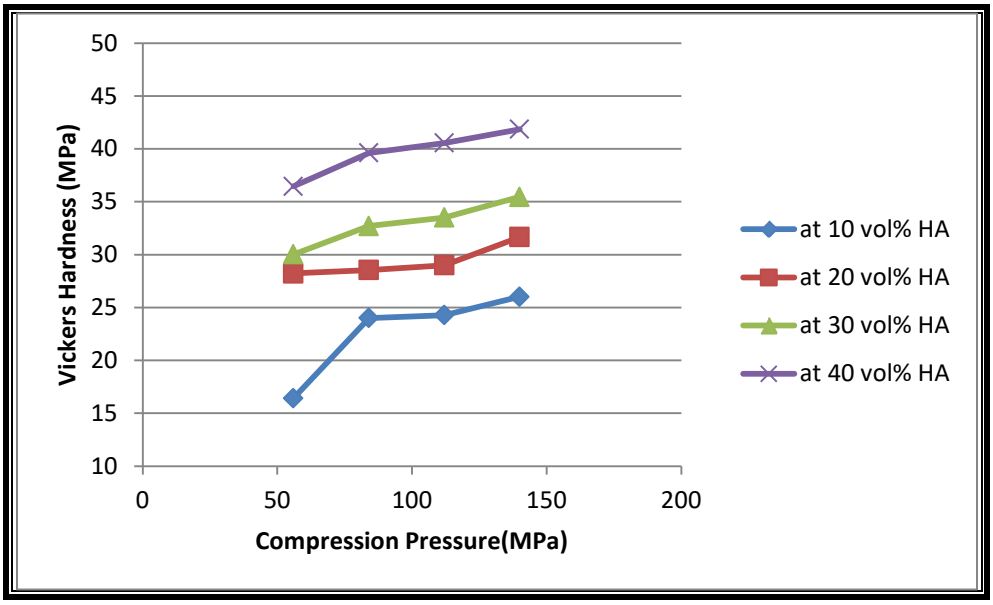


Figure (9) Effect of compression pressure on the Hardness at different vol% of HA.