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Original Research Article

Evaluation of Mechanical Properties of Zirconia Restorative Dental Materials Reinforced with Goat *(Capra hircus)* Bone

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ABSTRACT

A significant issue in dental health care services is the wear and fracture of restorative dental materials. This study investigated the properties of dental restorative materials made of resin-based zirconia composite reinforced with goat (Capra hircus) bone. The bone was calcined at three different temperatures: 750°C, 850°C, and 950°C. The bone's percentage content ranged from 0% to 20% for each calcination temperature, resulting in thirteen samples that were examined for wear rate, hardness, impact, tensile strength and microstructure. The results show that it is possible to create dental restorative samples with 15% to 20% Capra hircus bone at a calcination temperature of 850 °C. The bone and teeth have similar chemical compositions, making the bone a good raw material for the creation of dental restorative materials.

1. INTRODUCTION

The mouth is a delicate and sensitive mechanism of the general body structure that allows us to perform two primary functions: eating and speaking. The tooth is an operational part of the system. However, tooth loss is becoming a widespread concern in our immediate surroundings, which can be caused by tooth decay, cavities on the teeth, biting activities, food slurry, salivary factors, tooth brushing behaviours, periodontal sickness, contamination, etc. (Mandurino et al., 2023). This might cause discomfort and impair our overall well-being. These tooth issues are often repaired or replaced using materials that have the qualities anticipated of dental materials.

In recent years, several dental materials have been explored and created, such as glass ionomer cement, silicates, resin-based composite materials, and so on. These materials are predicted to withstand high compressive pressures in the mouth while also providing good aesthetic features. The most common dental restorative materials are resin-based composites (Chan et al., 2010). Furthermore, fillers with varying particle sizes have been included into these resin monomers to improve the mechanical, wear, and aesthetic aspects of dental materials.

Resin composites have recently gained popularity for tooth repair and restoration. Because of its great aesthetic qualities (Chan et al; 2010). Resin dental restorative composites have been shown to endure longer than dental amalgams, particularly in posterior restorations, which is why this study used resin as a binder and Zirconia as the main matrix.

Zirconia is a ceramic substance in powdered form. It is made up of zirconium and oxygen, having the chemical formula ZrO2. The crystalline microstructure of this material makes it ideal for application in dentistry due to its biocompatibility, low heat conductivity, corrosion resistance, and high tenacity (Ruben et al. 2014). Zirconia offers superior mechanical capabilities when compared to any other ceramic materials due to its distinct reinforcing features (Guazzato et al., 2014). Dental ceramics are among the most aesthetically pleasing and biocompatible restorative materials (Mohammed et al., 2022).

This aim of this research therefore, is to explore a raw material that integrates waste bio-ceramic material such as goat bone which is known scientifically as *Capra hircus* bone to serve as filler material, Zirconia as matrix and resin as a binder to produce high quality dental restorative material. Low Shrinkage, wear properties and good mechanical properties are expected of materials to be employed for dental restoration. These were considered while selecting these materials in order to produce a good dental restorative material that will revolutionize the dental industry.

2. MATERIALS AND METHODS

2.1. Material Collection and Preparation of Samples

The materials used in this study are shown in Figures 1a – 1e. The bio-ceramic filler used is *Capra hircus* bone. The bone was obtained at the Bariga local market in Lagos State, Nigeria. It was thoroughly cleaned with warm water before being soaked in hot water at 100 ºC for 6 hours. These criteria were chosen based on previous experience with pre-experimental activities. The bone was calcined with muffle furnace in Metallurgical and Materials Engineering Laboratory at University of Lagos. The calcination was done at three distinct temperatures: 750ºC, 850ºC, and 950ºC. The calcined bones were crushed and ground into a fine powder before being sieved to 150 microns (µ). The zirconia was obtained from a Chemical shop at Sango Ota in Ogun State, Nigeria.

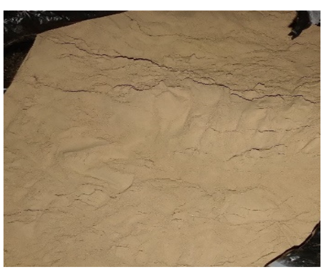

Figure 1a: Zirconia

Figure 1b: Calcined goat *Capra hircus*) bone

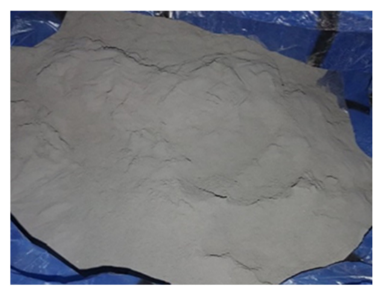

Figure 1c: Crushed bone

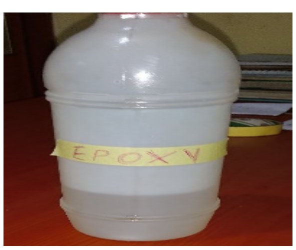

Figure 1d: Resin

Figure 1e: Weighing balance

The composite samples were made using the casting method, with a total blend weight of 350g, as the proportion of *Capra hircus* bone particle was adjusted throughout at 5 wt.% interval, with resin constant at 10 wt.% and zirconia the remaining percentage. Materials were weighed using an electric digital weighing balance. Based on the weight percentage of particle to be used in the resin. This procedure was performed for 13 weight fractions of particles required at (5%, 10%, 15%, and 20% of bone) for various temperatures, as given in Table 1. The slurry is then poured into moulds of various shapes and sizes, depending on the tests to be conducted, and allowed to set for 24 hours. Table 1 below shows the variation in the composition of samples produced in this research. The control sample tagged 'V' contains only 90% zirconia and 10% resin.

Table 1: Samples formulations and different calcination temperatures

S/N	Calcination temp (^oC)	Sample code	Zirconia (wt. %)	*Capra hircus* bone (wt. %)	Resin (wt. %)
1	None	V	90	0	10
2	750^{o}C	A1	85	5	10
3		A2	80	10	10
4		A3	75	15	10
5		A4	70	20	10
6	850^{o}C	B1	85	5	10
7		B2	80	10	10
8		B3	75	15	10
9		B4	70	20	10
10	950^{o}C	C1	85	5	10
11		C2	80	10	10
12		C3	75	15	10
13		C4	70	20	10

2.2. Chemical Composition Analysis

The chemical compositions of the raw materials were determined using X-ray Fluorescence (XRF) Analyzer (SKYRAY Instrument: EDX 3600B). This was carried out at National Geosciences Research Laboratory (NGRL), Kaduna.

2.3. Tensile Test

The tensile test was performed using a Universal Instron Machine. The tensile test specimen preparation and testing procedures were carried out in accordance with (ASTM D638-14), employing a dumbbell test piece. Each tensile specimen is placed in the Instron Universal Tester and then put under tension. As the specimen stretches, the computer provides a graph and all the necessary parameters up until the specimen breaks. The tester automatically plots a load versus extension graph and calculates the specimen's tensile strength, tensile strain, modulus, and tensile strain at break. The Test was carried out at Obafemi Awolowo University.

2.4. Impact Test

The impact energy of samples with various combinations of Zirconia and *Capra hircus* bone reinforced with resin were measured according to (ASTM D256, 2020) standard. The test samples were prepared and placed, after which the hammer was skillfully thrown and the readings from the pointer were recorded.

2.5. Wear Resistance Test

The wear rates of different samples at different loads and time were measured according to the (ISO 14569-1:2017). The pin on a disc method was used to determine the particular wear rate for samples made of zirconia and *Capra hircus* bone. Using a rotating speed of 125 rev/min and applied loads of 1.15 kg and 1.55 kg, the specific wear per sample was computed.

2.6. Brinell Hardness Test

The hardness test was performed in accordance with the (ASTM E10, 2023) standard. This was performed at the University of Lagos Metallurgical laboratory. The test samples were indented on the surface at various points, and the tester's display was used to calculate the average results.

3. RESULTS AND DISCUSSION

3.1. Chemical Composition

The X-Ray Fluorescence result of the materials are presented in Table 2. The major constituent of *Capra hircus* bone is CaO, this will enhance the bio-compatibility of the restorative material produced with the dental system. Zirconia contains high percentage of ZrO_2 as shown in Table 2, this improved the corrosion resistance and high tenacity of the dental restorative sample produced.

Table 2: XRF composition analysis of goat (*Capra hircus*) bone and zirconia

Oxide composition (%)	CaO	ZrO_2	SiO_2	Al_2O_3	P_2O_5	Fe_2O_3	LOI
Capra hircus	62.195	0.020	2.324	3.070	30.092	0.496	1.803
Zirconia	1.50	63.293	22.242	2.472	2.187	3.242	1.53

3.2. Tensile Test

The results of tensile strength of varying percentage of the dental restorative materials are presented in Figure 2. It could be seen that B3 (75% zirconia, 15% *Capra hircus* bone, 10% resin) yields the optimal strength of 7.69 MPa, followed by C1 (85% zirconia, 5% *Capra hircus* bone, 10% resin) at 5.80MPa and C4 (70% zirconia, 20% Capra hircus bone and 10% resin) at 5.82 MPa whereas the B4 (70% zirconia, 20% Capra hircus bone and 10% resin) has the lowest strength of 0. 95 MPa. From observation, the tensile strength increased consistently from V (90% zirconia, 0% Capra hircus bone, 10% resin) at 3.43MPa up to B1 (85% zirconia, 5% *Capra hircus* bone, 10% resin) at 5.52 MPa. The increase in strength from 3.34 to 7.69 MPa represents a steady but substantial improvement in tensile strength. This could be attributed to proper stress propagation or distribution, which is required for superior composite performance under tensile loading. Samples B3 and C1 have superior tensile strength compared to others. This established that *Capra hircus* bone improved the tensile strength of dental restorative samples more than other combinations and is comparable to that of a real tooth.

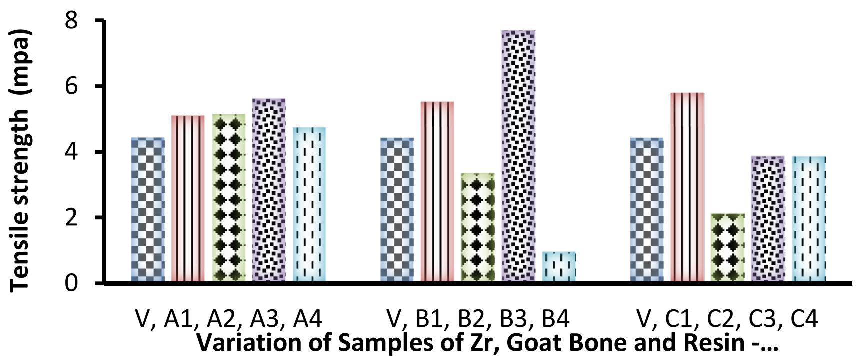

Figure 2: Tensile strength of the composites of zirconia, *Capra hircus* bone and epoxy resin calcinated at 750°C, 850°C, 950°C

3.3. Impact Test Result

The impact energy of various combinations of Zirconia and goat bone reinforced with resin are presented in Figure 3. The results show that A2 (80% zirconia, 10% *Capra hircus* bone and 10% resin), recorded the highest impact toughness of 8.22J followed by B4 (70% zirconia, 20% *Capra hircus* bone, 10% resin) at 7.37J. With changes in the calcination temperature at 850°C, a steady increase in impact energy was observed from B1 (85% zirconia, 5% *Capra hircus* bone, 10% resin) which has the lowest value of 5.19J to B4 which has the value of 7.37J with changes in the filler variation on samples. The values obtained from samples calcinated at 750°C and 950 °C were fluctuating showing instability. The above results indicate that B4 (70% zirconia, 20% *Capra hircus* bone, 10% resin) calcined at 850°C absorbs enough energy before failure.

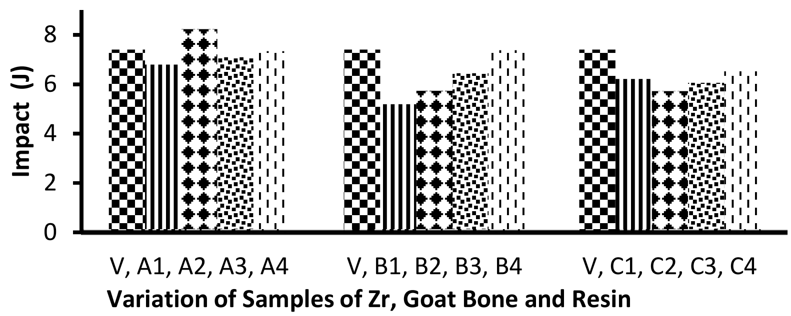

Figure 3: Showing the impact energy of the composites of zirconia, *Capra hircus* bone and epoxy resin calcinated at 750ºC, 850ºC and 950ºC.

3.4. Wear Resistance Test Results

The wear rates of different samples at different loads and time were measured and the results are presented in Figures 4a and b. It was observed that the addition of Zirconia and *Capra hircus* bone generally reduced the wear rate in other words, it increased the wear resistance. In Figure 4a using Load 1 (1.15kg) and speed of 250 RPM at 30 seconds, it was observed that B2 (80% zirconia, 10% goat bone and 10% resin) and A1 (85% zirconia, 5% goat bone, 10% resin) have the highest wear rate of 0.0058gm/m and 0.0056gm/m respectively.

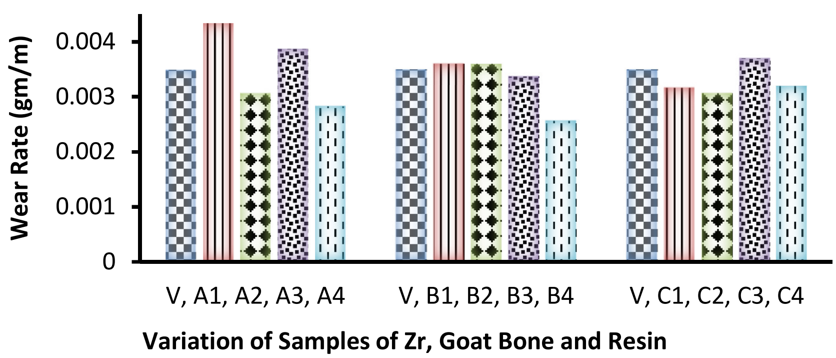

Figure 4a: Wear rate of the composite of zirconia, *Capra hircus* bone and epoxy resin calcinated at 750ºC, 850ºC, 950ºC. using load 1.15kg

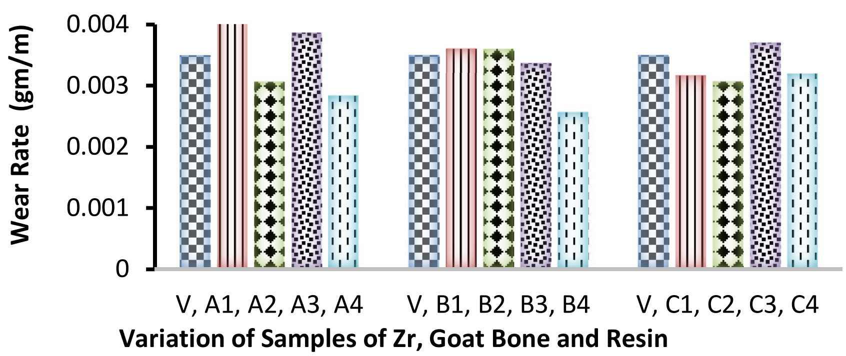

Figure 4b: Wear rate of the composite of zirconia, *Capra hircus* bone and epoxy resin calcinated at 750ºC, 850ºC, 950ºC. at load of 1.55kg

At 90 seconds, B4 (70% zirconia, 20% goat bone, 10% resin), recorded the lowest wear rate of 0.0023gm/m. In Figure 4b using Load 2 (1.55kg) for 30 seconds, C3 and A1 also recorded the highest wear rate of 0.0061gm/m and 0.0059gm/m respectively. B4 (70% zirconia, 20% goat bone, 10% resin) at 90 seconds and B3 (75% zirconia, 15% goat bone and 10% resin) at 60 seconds maintained the lowest wear rate. The results in Figure 4b indicate that B3(75% zirconia, 15% goat bone and 10% resin) at 60 seconds and B4 (70% zirconia, 20% goat bone, 10% resin) at 90 seconds have suitable wear resistance. The relationship found between the wear behavior and the hardness properties is in line with the study by Morozova et al., (2016).

3.5. Brinell Hardness Test Result

The hardness test was performed in accordance with the (ASTM E10, 2023) standard, and Figure 5 displays the results of the hardness values of the samples. The results revealed that C4 (80% Zirconia / 20% *Capra hircus* bone calcined at 950 0C) had the maximum hardness value of 29.3HV, whereas A1 (85% zirconia, 5% *Capra hircus* bone, 10% resin) had the lowest hardness value. Overall, the results followed the same trend, with an initial hardness of 20.2HV at V (pure epoxy resin) and a subsequent decline to 16.5HV at A1 (85% zirconia, 5% *Capra hircus* bone, 10% resin). It eventually climbed to 23.6 HV at A3 (75% zirconia, 15% *Capra hircus* bone, and 10% resin). It reduced again at B1 (85% zirconia, 5% *Capra hircus* bone, and 10% resin), after which it continued to rise in hardness. The results show that B4 has a moderate hardness value of 20.4Hv, which is comparable to a natural tooth. Samples with a high hardness value are fragile and readily fractured, whereas samples with a low hardness value cannot withstand indentation. This result is in agreement with Stencel et al., (2018).

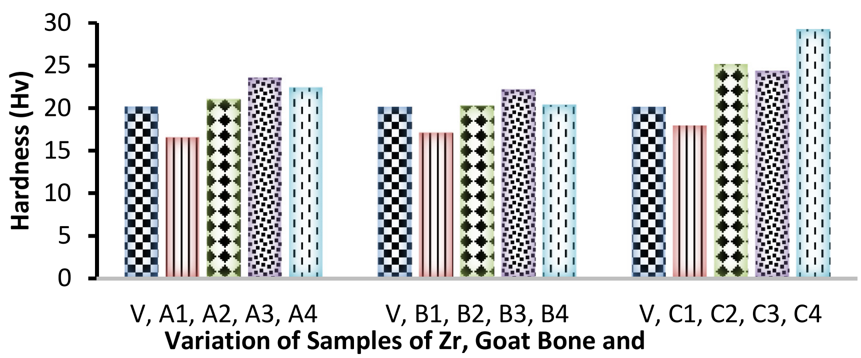

Figure 5: Hardness of the composite of zirconia, *Capra hircus* bone and epoxy resin calcinated at 750°C, 850 °C, 950 °C.

3.6. X-Ray Diffractometry (XRD) Analysis

The X-ray diffractometry analysis results shown in Figures. 6a – 6g, revealed that majority of the samples showed distortion in the peak of the tetragonal phase, widening after the *Capra hircus* bone fillers were introduced into the zirconia samples. The control sample showed a sharp peak at Bragg angle of 35° indicating crystalline nature of the sample. All the samples with additives calcined at 750°C, 850°C and 950°C followed almost the same pattern with one another. They showed slight broad peak between 30° – 37° Bragg angle and between 37° – 40°, a very sharp peak appeared indicating crystalline phase. At Bragg angles above 40°, the peak disappeared indicating increase in amorphous phase. This trend was in conformity with the study carried out by (Zhang. et al., 2018; Okoubulu et al., 2023).

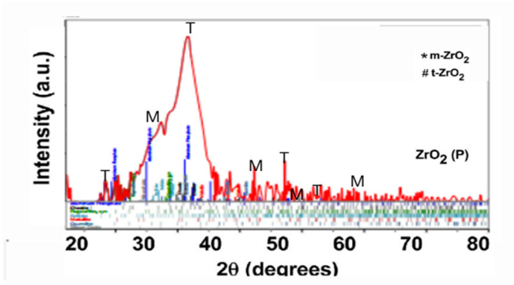

Figure 6a: XRD of sample V

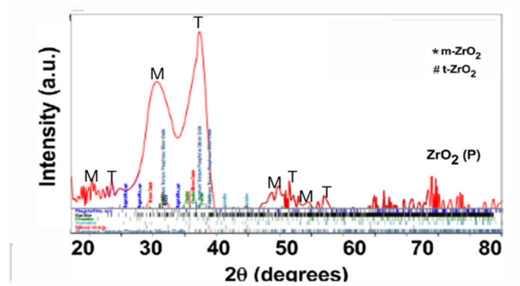

Figure 6b: XRD of sample A2

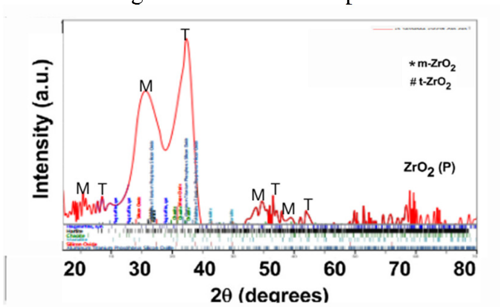

Figure 6c: XRD of sample A3

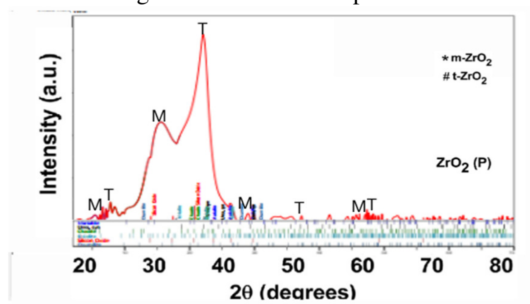

Figure 6d: XRD of sample B2

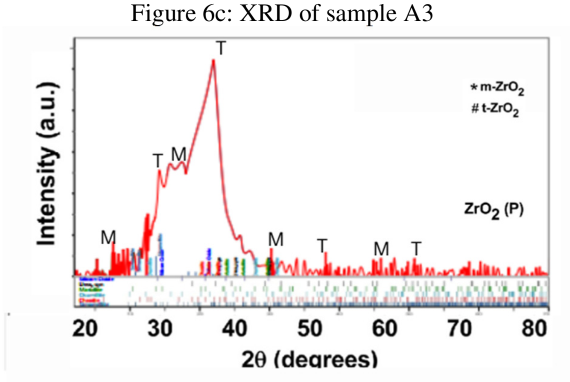

Figure 6e: XRD of sample B3

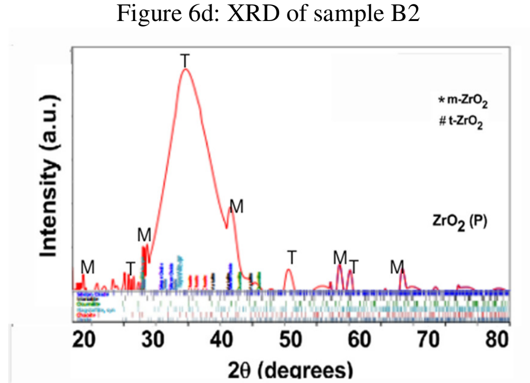

Figure 6f: XRD of sample C2

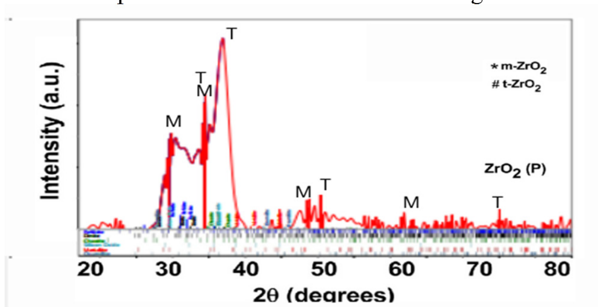

Figure 6g: XRD of sample C3

3.7 Scanning Electron Microscopy (SEM)

SEM analyses for samples V (control sample without *Capra hircus* bone), A2 (80% zirconia, 10% bone at 750ºC and 10% resin), A3 (75% zirconia, 15% bone at 750ºC, 10% resin), B2 (80% zirconia, 10% bone at 850ºC and 10% resin), B3 (75% zirconia, 15% bone at 850ºC and 10% resin), and C2 (80% zirconia, 10% bone at 950ºC and 10% resin) and C3, are shown in Figures 7a – 7g. The micrographs of the samples confirmed what was obtained in the mechanical properties results showed earlier. The surface morphologies of the samples revealed that the control sample without any additive has a better uniformity indicating absence of different phases. In other samples, the presence of filler materials which have significant effect on the mechanical properties of the samples showed presence of different phases (Figures 7b -7g). This can be attributed to the addition of Capra hircus bone which improved the mechanical properties of the samples as indicated in the results obtained earlier. The results followed the same trend with the study carried out by (Ouyang et al., 2011; Guazzato et al., 2014).

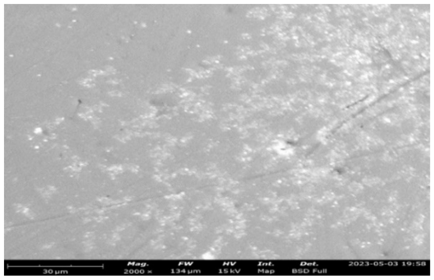

Figure 7a: SEM Analysis of sample V

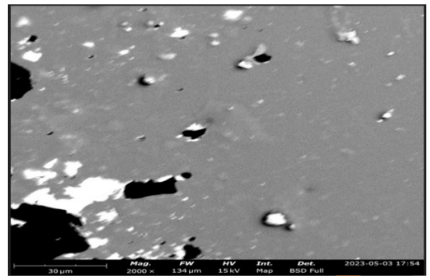

Figure 7b: SEM Analysis of sample A2

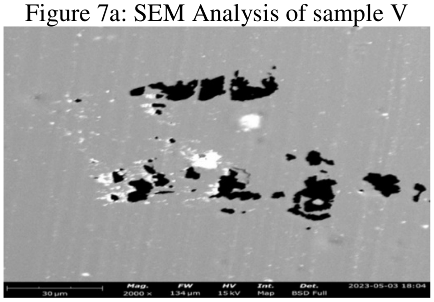

Figure 7c: SEM of A3

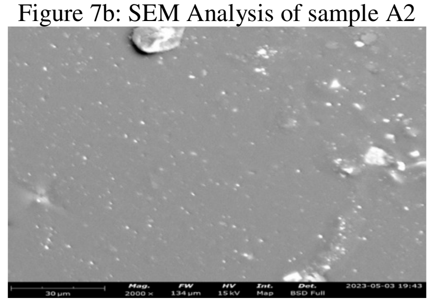

Figure 7d: SEM Analysis of sample B2

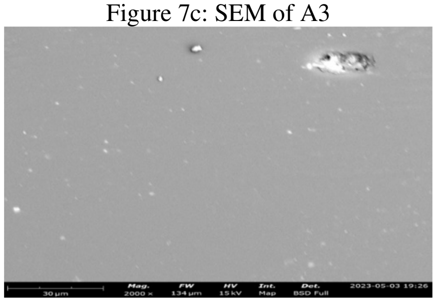

Figure 7e: SEM B3

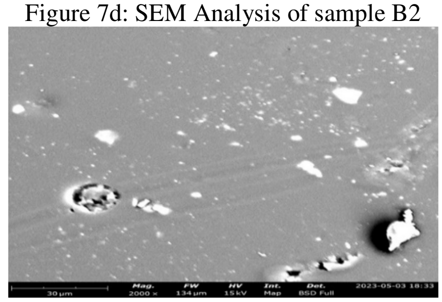

Figure 7f: SEM Analysis of sample C2

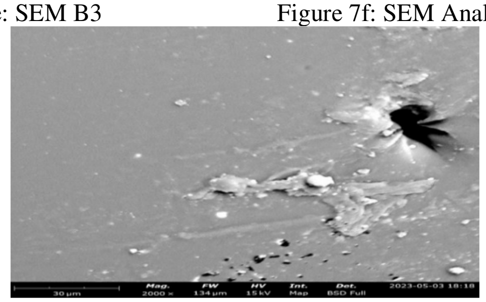

Figure 7g: SEM of C3

4. CONCLUSION

In this study, dental restorative samples have been produced from zirconia and *Capra hircus* bone mixed with resin using casting method. The effects of the various compositions of the additives and calcination temperatures on the properties of the dental restorative samples were observed. The impact energy of various combinations of zirconia and goat bone mixed with resin revealed that A2 (80% zirconia, 10% *Capra hircus* bone, and 10% resin) had the maximum impact toughness of 8.22J, followed by B4 (70% zirconia, 20%

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Capra hircus bone, 10% resin) at 7.37J. The wear rate results show that B3 (75% zirconia, 15% *Capra hircus* bone, and 10% resin) at 60 seconds and B4 (70% zirconia, 20% *Capra hircus* bone, 10% resin) at 90 seconds have adequate wear resistance, with B4 having the lowest wear rate of 0.0023gm/m. B4 also has a moderate hardness value of 20.4 Hv, relative to a natural tooth. Therefore, from the study, it can be established that a dental restorative material with suitable properties can be produced using 70 - 75 wt.% of zirconia, 15 - 20 wt.% of *Capra hircus* bone calcined at 850°C and 10 wt.% of resin.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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