International Information and Engineering Technology Association
Advancing the World of Information and Engineering

Journal Européen des Systèmes Automatisés
Vol. 58, No. 5, May, 2025, pp. 887-894
Journal homepage: http://iieta.org/journals/jesa

Agro-Wastes Materials as a Novel Approach for High-Speed Steel (HSS) Tool Case-Hardening

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https://doi.org/10.18280/jesa.580502

Received: 10 March 2025
Revised: 16 April 2025
Accepted: 26 April 2025
Available online: 31 May 2025

Keywords:
agro-waste, case-hardening, cutting tool, machining, steel, SDG, waste recycling

ABSTRACT

The goal of this study was to create a cutting tool out of carbonized snail shells and cow horns, two agricultural waste products. The materials were chosen according to their chemical compatibility, material qualities, and required temperature and pressure. Energy dispersive X-ray fluorescence (XRF) compositional analysis showed that calcium oxide (CaO) is the main component of snail shells, whereas silicon dioxide (SiO_2), ferric oxide (Fe_2O_3), calcium oxide, and sulfur trioxide (SO_3) are present in significant amounts in cow horns. Cutting temperature, surface roughness, and material removal rate (MRR) were evaluated as part of the cutting tool performance evaluation, which was based on a mix ratio of these materials. When compared to untreated samples and other mix ratios, the snail shell to cow horn mix ratio of 70-30% showed the best results, with lower cutting temperatures, smoother surface finishes, and higher MRR.

1. INTRODUCTION

Manufacturing is simply the process of turning a raw material or part into finished product [1]. Das [2] reported that variety of tools are essential for turning raw materials into a finished good fit for human consumption. This would then require the need for a cutting tool in other to shape materials. Different sizes and shapes of raw materials are delivered for machining and fabrication - so most operations require some kind of cutting activity in order to make a workpiece because raw material dimensions rarely match those required by the workpiece [3]. Hand tools were used to cut and shape materials for the creation of commodities including cooking utensils, carriages, ships, furniture, and other products prior to the Industrial Revolution of the 18th century [4, 5]. Figures 1 (a-c) shows the development of hand tools in early times as they were progressively refined through the ages.

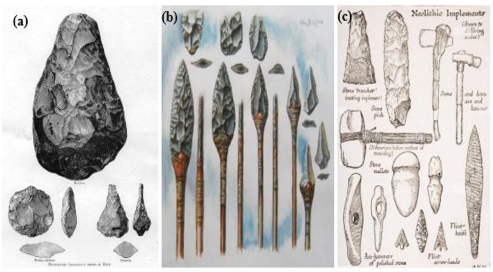

Figure 1. Development of cutting tools during pre-historic times (a) Paleolithic age (b) Mesolithic age (c) Neolithic age [6]

The Neolithic revolution cleared way, transitioning into the bronze and iron ages for the use and development of various metals that could be found – due to their inherent mechanical properties [7]. Research on manufacturing is currently primarily focused on increasing productivity, which means materials that can sustain higher cutting loads than the current attainable machining limits are being developed, particularly for cutting tools [8]. Early humans found that by combining copper with tin or copper with brass, they could create strong, durable metals [9, 10]. Unaware of the fact that they were exploiting the process which is known currently as solid solution hardening or the alloying process, which is essentially the act of dissolving one metal into another [9].

Mills [11] discussed about the development of cutting tool materials in which he classified them into; high speed steels, cemented carbides, ceramic – tougher materials including, alumina-based composites, silicon nitrides, cubic boron nitride, and diamond – as they progressively get stronger. Then there is the non-contact mechanical-based cutting – laser and water jet cutting, which has revolutionized the cutting industry since its development in the 1960's [12] for laser cutting, and in the 1930's [13, 14] for water jet cutting; offering a number of benefits over mechanical cutting, including increased accuracy, efficiency, and environmental friendliness [15].

The current trend among researchers today focuses on how agricultural waste materials can be used for sustainable green production and other applications to industries, of one which is with the steel industry. Ogedengbe et al. [16] reviewed on how agro-waste can be a sustainable source for steel reinforcement – highlighting the use of high-quality carbon derived from these agro-waste materials for steel reinforcing.

Olorunyolemi et al. [17] worked on enhancing mechanical behavior and grain characteristics of aluminium matrix composites by incorporating agro-waste materials. Overall, the advantages of utilizing agro-waste in industrial sectors extend beyond economic benefits to include environmental stewardship, resource efficiency, cheaper, easily accessible and supports sustainability.

Previously, attempts have been made to improve on the strength of various steel types. Table 1 shows some agro-waste materials that have been employed to improve particular steel types.

Table 1. Agro-waste materials used for steel enhancement

Agro-wastes	Steel	Method Adopted	References
Palm Kernel Shell	High Speed Steel	Pulverization Heat Treatment	Afolalu et al. [18]
Coconut Shell, Rice Husk, Cow horn	Aluminium Metal Matrix	Mixed Methods	Joseph and Babaremu [19]
Rice Husk	Aluminium Metal Matrix	Liquid Metallurgy	Saravanan and Kumar [20]
Walnut Shell	Alloy Steel	Stir Casting, Heat Treatment, Machining	Akande et al. [21]
Coconut Shell	Aluminium 7075	Stir Casting, Heat Treatment	Kubendiran et al. [22]

The metal cutting tool's wear is a complicated phenomenon with numerous working wear mechanisms. But the general issue with the removal process of a material is the difference in hardness between the tool and the material – which one of the two materials would have a lower wear resistance than the other. This problem is being investigated by current researchers, along with the possibilities of how agro-waste can be utilized for this purpose – thereby addressing the issue of poor waste management on the environment.

2. MATERIALS

2.1 Material acqusition

(1) Snail Shell:

Cleaning & washing – snail shell was washed with soap, water and sponge to thoroughly remove slime and dirt from the inside of the shell (Figure 2a).

Drying – snail shell was then sundried after washing to lower its moisture content, since excessive levels of moisture can interfere with combustion and introduce undesirable elements into the heat-treating environment. Mechanical techniques, making use of an oven is also a substitute for drying of the snail shell. The snail shells were sun dried for 72hrs but were not oven-dried for reasons being that snail shells are not hydroscopic materials, meaning they do not readily absorb moisture from the air because they mostly contain calcium carbonate. Biological materials like egg shells containing calcium carbonate possesses moisture content of 1 to 5 percent after being sundried.

Grinding – to increase the waste materials effectiveness as heat-conducting substrates, both agro-waste materials would go through a grinding, sieving and carbonization process. The snail shell was crushed with a pistol and mortar, to drastically reduce the size as shown in Figure 2b; and the blended with an industrial blender to further reduce the size as shown below in Figure 2c.

Sieving – grinded snail shell was then sieved to guarantee a more uniform sized distribution by separating smaller from larger particles. This may help ensure that carbon diffuses uniformly throughout the case hardening process. Snail shell was sieved with to a minimum size of 300 microns with a lab standard sieve as shown in Figure 2d.

Carbonization – snail shell was then placed in furnace to control the heating conditions as shown in Figure 2e. The furnace was then sealed and the snail shell was heated gradually to elevated temperatures at about 400℃ for 2hrs as shown below in Figure 2c. This temperature choice was guided by previous works of Daud et al. [23] and Li et al. [24].

Sealing of the chamber was necessary to reduce the entry of oxygen, preventing the materials from burning completely [25]. An inert atmosphere in the furnace was ensured to be certain of a uniform carbonization of the steel.

After carbonization, the snail shell was then removed from the furnace as shown in Figure 2f and then collected in a zip bag in order for it to be sealed, preventing the snail shell from absorbing moisture.

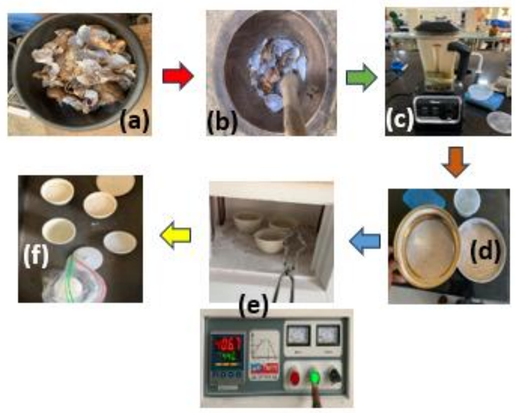

Figure 2. Snail shell preparation cycle (a) washing (b) milling (c) grinding (d) seiving (e) carbonizing (f) packaging

(2) Cow Horn:

Cleaning – the cow horn was obtained dry from the abattoir therefore it would be counterproductive to wash it with water, drastically increasing its moisture content. Therefore, the cow horn wan cleaned to get rid of dirt from the inside and on the surface (Figure 3a).

Drying – after milling, cow horn was then sundried firstly to lower its moisture content shown in Figure 3b – since excessive levels of moisture can interfere with combustion and introduce undesirable elements into the heat-treating environment; mechanical techniques, making use of an oven to further reduce the moisture content was later used for drying of the cow horn – at 120℃ for 24hrs.

Grinding – the cow horns were grinded with a milling machine to remove chips of cow horn at the initial stage of grinding as shown in Figure 3c below; in which they were then grinded with an industrial blender to further reduce their sizes at the final stage of grinding.

Burning – before the carbonization of the cow horns, the horns were first burnt because of its dust like nature in grinded form. The cow horn was burnt in a pan on a gas cooker as shown in Figure 3f.

After burning, the solidified cow horn was then crushed again with a mortar and a pistol to obtain its char before sieving.

Sieving – the obtained cow horn ash was then sieved to guarantee a more uniform sized distribution of the agro-waste just like it was done for the snail shell. Cow horn ash was sieved to same size as the snail shell, to a minimum size of 300 microns with a lab standard sieve (Figure 3e).

Carbonization – the cow horn was also placed in a furnace just like the snail shell, to control the heating conditions as shown in Figures 3f. Same as the snail shells, the cow horn was also and heated gradually to elevated temperatures – at about 400°C for 2hrs.

Figure 3. Cow horn preparation cycle (a) cleaning (b) drying (c) grinding (d) blending (e) sieving (f) carbonizing (g) packaging

2.2 Equipment

The equipment used for the study includes a Muffle electric furnace with model number STM-3-17, a rated voltage of 200V and a capacity of 1700°C for the carbonization of both materials. A commercial blender (model FD-990) with a rated voltage of 220 – 230V and a capacity of 2200W was employed for grinding of both materials. A precision electronic balance (model XY20002C), with a model number of 2011911062, and a capacity of 2100g was used for the weighing of both agro-waste samples. A milling machine was employed for the chipping of cow horn. An X-ray fluorescence (XRF) spectrometer was also used for the characterization of both agro-waste materials. An angle grinder and arc welding machine for the forming of the steel housing.

Figure 4. Model of the steel housing/packing box

2.3 Methods

2.3.1 Experimental procedure

Cutting tools was inserted into a steel container (Figure 4) after which the obtained agro-waste ash was packed around the cutting tool and sealed to guarantee sufficient coverage throughout the heat treatment to enable efficient carbon transfer.

A. Design for fabrication of steel housing

The amount of agro-waste (cow-horn) had an unexpectedly drastic reduction in size after the carbonization process. Therefore, the area of the tool to be heat treated, had to be reduced in other for the steel housing to contain the required mixing ratio. A model showing the design of the steel housing is shown in Figure 5, where fusion 360 was used to calculate the total volume needed for the agro-waste to fill the packing container while the tool is submerged in the container. This was done by subtracting the volume of the tool from the volume of the container (both volumes were given by the software).

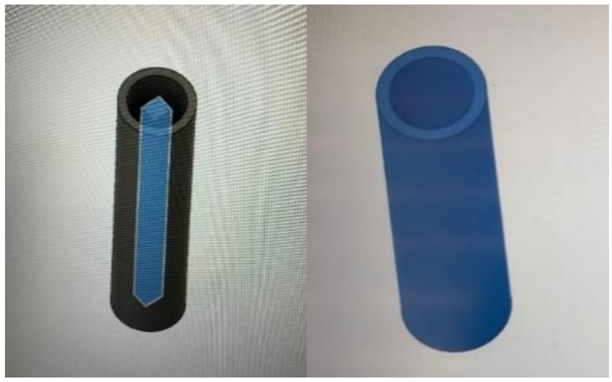

Figure 5. Calculation of fill volume (a) volume of tool (b) volume of steel housing

The design was then made to account for the loss of agro-waste material during carbonization by restricting the heat treatment (case hardening) area to only a length of 5cm. therefore, with the fill volume known, it becomes easier to calculate the amount of agro-waste needed for both mixing ratios – mix ratio A, 90-10% and mix ratio B, 70-30%. Table 2 shows the calculation of the required volumes Eqs. (1) and (2).

Table 2. Mixing ratios of the cow horn and snail shell

	Snail Shell	Cow Horn
Mix ratio A	$\frac{90}{100} * 33.132$	$\frac{10}{100} * 33.132$
Mix ratio B	$\frac{70}{100} * 33.132$	$\frac{30}{100} * 33.132$

The table shows the equations for the mixing ratios of both samples with snail shell taking 90 & 70% of the mixture and cow horn taking 10 & 30% of the mixture.

After the amount (in volume) of agro-waste material was calculated, both samples were then measured, the amount of snail shell was measured using a measuring cylinder and the volume of the cow horn was measured using the weight. After which the samples were mixed with their various mixing ratios and packed into the steel housing – the top of the housing was then sealed with foundry clay.

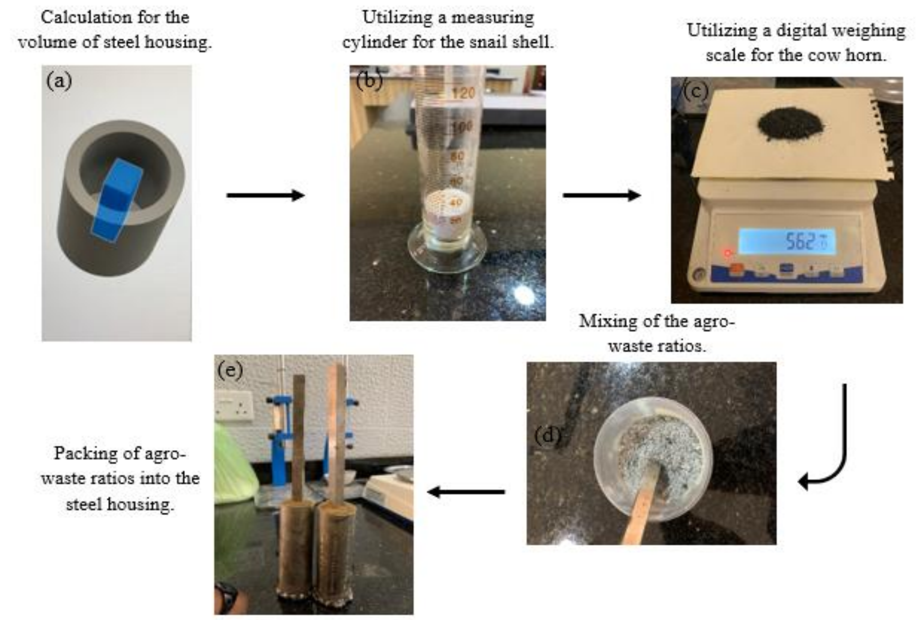

Figure 6. Packing of agro-waste into casing (a) amount of carburizer (b) amount of snail shell (c) amount of cow horn (d) mixing of both agro-waste (e) packed agro-waste in steel housing

B. Heat treatment (case hardening) process

The materials with their mixing ratios were packed into the steel housing, would be able to withstand the temperature (700 degree C). The stainless steel used is a low carbon version of 316 – with a thickness of 0.5cm, which is used for applications like welded structures, marine applications, chemical processing etc. also offering good corrosion resistance after welding. With a 0.5 thickness and properties of its grade, it would be a reliable fit for its application – with a melting point of 1370 – 1400°C.

The HSS tool was grinded to a facing tool for the turning operation before inserting into the steel housing and casing was sealed with foundry clay, before heating at 700°C for 4hrs as shown in Figures 6 and 7. The HSS tool was then quenched in a mineral based oil (SAE 15W-40) and tempered in an oven at 200°C for 2hrs.

Figure 7. Case hardening of both samples (a) foundry clay for case sealing (b) heating reactor (c) heating at 700°C

2.3.2 Machining parameters and responses

Figure 8 displays the machining process (turning operation) of a low-carbon steel rod with the casehardened tools – also shows the temperature reading of the cutting tool with the use of a thermometer (k-type thermo-couple) during machining at different speeds, feeds and depths.

2.3.3 Design of experiment

The experiment was designed using Minitab version 18.0. the three factors selected are cutting speed, feed rate and depth of cut. Taguchi Method was used to design the experiment with a total of 9 runs. Table 3 and Table 4 below, shows the machining parameters with levels and the L9 orthogonal array respectively. Literature, machining capacity, and initial trial machining results served as a guide for selecting the machining parameters.

Figure 8. Machining to test viability of cutting tool

Table 3. Machining parameters and levels

Control Factors	Levels		
	1	2	3
Cutting speed (rpm)	600	800	1000
Feed rate (mm/min)	8	9	10
Depth of cut (mm)	0.01	0.007	0.005

Table 4. Orthogonal array for experiment

Turning Run(s)	Cutting Speed (rpm)	Feed Rate (mm/min)	Depth of Cut (mm)
1	600	8	0.01
2	600	9	0.007
3	600	10	0.005
4	800	8	0.01
5	800	9	0.007
6	800	10	0.005
7	1000	8	0.01
8	1000	9	0.007
9	1000	10	0.005

2.3.4 Temperature measurement

A digital thermometer (K-type thermocouple) was used to capture the temperature. This was done by setting the probe as close as possible to the cutting zone for the duration/length of feed. The temperature measurement was captured in triplicates and an average was found to ensure exactness of measured temperatures.

2.3.5 Surface roughness, Ra

The surface roughness, Ra (μm), of the machined work piece was estimated using analytical methods based on process parameters for a turned surface. For turning process, the formula used is stated in Eq. (1) below:

$$Ra = \frac{f^2}{32r} \quad (1)$$

where, 'f' if the feed rate and 'r' is the nose radius of the cutting tool.

2.3.6 Material removal rate, MRR

The measurement of how much material was removed would also aid in testing for viability of the casehardened tool. For a turning process, the formula used to estimate/obtain the MMR during machining is stated in Eq. (2) below:

$$MRR = Spindle\ Speed * Feed\ Rate * Depth\ of\ cut \quad (2)$$

3. RESULTS AND DISCUSSION

3.1 Performance evaluation

3.1.1 Pre-experimentation analysis – carbonized agro-waste

Snail Shell

Table 5 shows the result of the compositional analysis of the snail shell sample using the energy dispersive X-ray fluorescence (XRF) analysis. The result confirmed the presence of the elements as stated in the first column (component) as the major constituents of the snail shell sample. The snail shell's XRF analysis reveals that calcium oxide (CaO), which makes up roughly 93.879 weight percent of the sample, is the main constituent. This is to be expected since the main material of snail shells is calcium carbonate ($CaCO_3$), which, when heated, breaks down into CaO. Significantly smaller amounts of silicon dioxide (SiO_2), iron (III) oxide (Fe_2O_3), and aluminum oxide (Al_2O_3) have also been found. Additionally, present are trace elements like cobalt, nickel, and copper, which could suggest interactions with the environment or dietary influences on the snail. Certain elements, such as phosphorus, sulfur, titanium, molybdenum, and chromium, are either absent or present in very small amounts in the sample.

Cow Horn

Table 6 shows the compositional analysis of the cow horn sample using the same methods as stated for the snail shell. The XRF results shows that the main ingredients of cow horn are silicon dioxide, ferric oxide, calcium oxide, sulfur trioxide, and magnesium, potassium, aluminum, zinc, and chlorine, all of which contribute significantly. These elements point to a robust and complex structure for the horn, with different elements contributing to its mechanical strength, stiffness, and biological functionality. A significant amount of keratin and other protein structures, which are typical of biological materials like cow horns, are indicated by the high concentrations of calcium and sulfur. The major and minor oxide components of the cow horn are listed in this table, along with the intensities of the important elements found during analysis. Although the presence of SO_3 may not be directly related to the improvement of strength of the steel by the cow horn, the presence of calcium oxide CaO (19.4%) could account for the eventual grain refinement which could result in the improvement of mechanical properties such as strength and elasticity of the steel.

Table 5. XRF analysis on snail shell particles

Component	Conc. (wt.%)	Error (wt.%)	Conc. (Mole%)	Error (Mole%)
SiO	0.279	0.260	0.266	0.249
V_2O_5	0.048	0.023	0.015	0.007
Cr_2O_3	0.000	0.000	0.000	0.000
MnO	0.006	0.010	0.005	0.008
Fe_2O_3	0.184	0.018	0.066	0.007
CoO	0.082	0.011	0.063	0.008
NiO	0.027	0.007	0.021	0.005
CuO	0.041	0.009	0.030	0.006
Nb_2O_5	0.114	0.438	0.025	0.095
MoO_3	0.000	0.000	0.000	0.000
WO_3	0.018	0.039	0.004	0.010
P_2O_5	0.000	0.000	0.000	0.000
SO_3	0.000	0.000	0.000	0.000
CaO	93.879	0.490	96.098	0.501
MgO	0.181	6.494	0.258	9.250
K_2O	0.000	0.000	0.000	0.000
BaO	0.072	0.072	0.027	0.027
Al_2O_3	3.995	1.172	2.249	0.660
Ta_2O_5	0.037	0.035	0.005	0.005
TiO_2	0.000	0.000	0.000	0.000
ZnO	0.006	0.008	0.004	0.005
Ag_2O	0.000	0.000	0.000	0.000
Cl	0.371	0.031	0.601	0.050
ZrO_2	0.141	0.108	0.066	0.050
SnO_2	0.520	1.181	0.198	0.450

Table 6. XRF analysis on cow horn particles

Component	Concentration (wt.%)	Error (wt.%)	Intensity (c/s)
SiO_2	12.381	1.859	5.787
Fe_2O_3	17.528	0.199	12.260
SO_3	22.909	0.972	9.175
CaO	19.418	0.493	13.878
MgO	2.110	34.654	1.273
K_2O	3.023	0.266	2.510
Al_2O_3	8.825	5.331	4.671
ZnO	4.666	0.090	3.749
Cl	6.390	0.355	6.390
MnO	0.374	0.053	0.289
CuO	0.859	0.055	0.686
TiO_2	0.865	0.113	0.519
V_2O_5	0.038	0.074	0.021
Cr_2O_3	0.072	0.053	0.049
Nb_2O_5	0.078	0.035	0.054
MoO_3	0.031	0.034	0.021
WO_3	0.122	0.327	0.097
P_2O_5	0.039	0.598	0.017
Ag_2O	0.052	0.096	0.049
ZrO_2	0.036	0.032	0.027
Ta_2O_5	0.065	0.198	0.053

3.1.2 Machining results

Table 7 displays the summary of results obtained from the machining process, test carried out and calculations made –

therefore, the follow can be deduced form the table.

Cutting Temperature

Cutting temperature increases as the spindle speed increases, it can be seen that the lowest cutting temperature ranges between the values in the 70-30% mix ratio (Table 5). At a speed of 600rpm, the untreated sample had a cutting temperature of 138℃, which was found to be the highest as opposed to the 70-30% mix ratio, with a cutting temperature of 131℃ displaying a better performance in thermal resistance. The temperature then increased again as the tool with the 90-10% mix ratio sample was used – increasing to 134℃, higher than the 70-30% mix ratio sample but lower than the untreated sample – displaying a better thermal resistance than the untreated sample but not more than the 70-30% mix ratio sample. Similar results also for the spindle speeds of 800 and 1000rpm. This shows that the 70-30% mix ratio had the highest thermal resistance as evident to its low temperatures during machining (Figure 9).

Table 7. Summary of result for machining parameters

	Spindle Speed	Cutting Temp (℃)	Surf. R (Ra)	Material Removal Rate (mm^3/mm)
Untreated sample	600	138	0.3500	48.400
	800	151	0.4870	50.920
	1000	178	0.6525	50.500
70-30% mix ratio	600	131	0.3033	50.800
	800	141	0.4244	53.440
	1000	169	0.6025	52.790
90-10% mix ratio	600	134	0.3233	49.360
	800	146	0.4580	51.400
	1000	173	0.6300	51.620

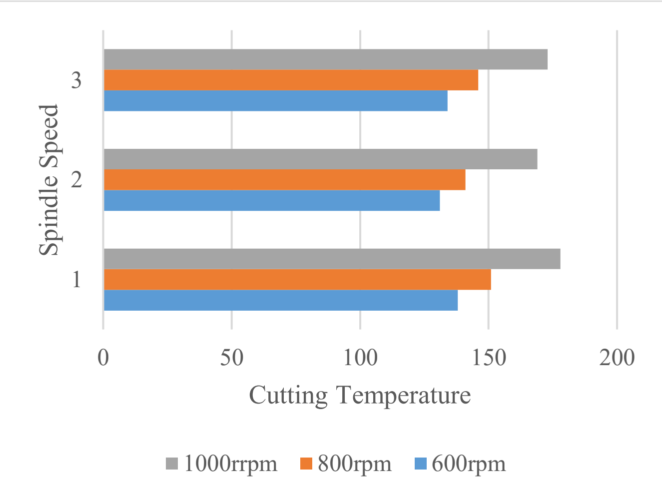

Figure 9. Spindle speed vs. cutting temperature

Surface Roughness

The lower the roughness average value, the smoother the surface. As the feed rate drops, the roughness average also decreases (Figure 10), with the 70-30% mix ratio showing the lowest Ra values. Compared to the 70-30% mix ratio, where a Ra of 0.30 mm demonstrated a better performance in surface finish, the untreated sample had the highest Ra of 0.35 mm at a feed rate of 8 mm/min. The tool with the 90-10% mix ratio sample was then used, and the Ra increased once more. This time, it increased to 0.32 mm, which was higher than the 70-30% mix ratio sample but lower than the untreated sample. This indicates that the surface finish of the tool was better than the untreated sample, but not more so than the 70-30% mix ratio sample. Comparable outcomes were noted for feed rates of 10 and 9 mm/min. This demonstrates that, as shown by its low Ra values during machining, the mix ratio of 70-30% had the best surface finish.

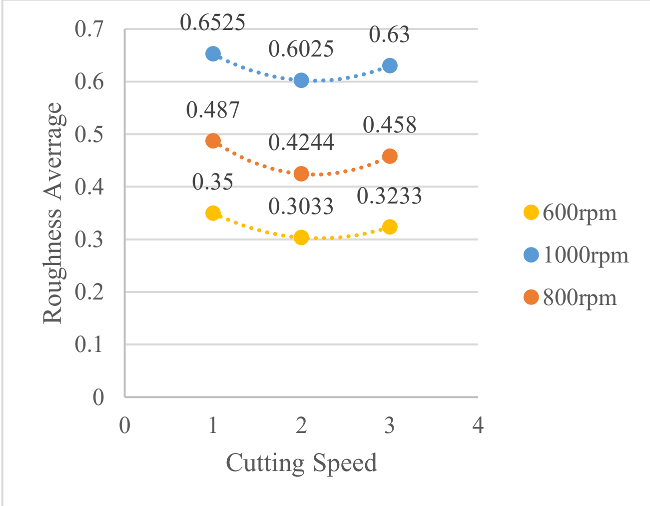

Figure 10. Roughness average vs. cutting speed

Material Removal Rate

The material removal rate (MRR) declines with decreasing depth of cut (Figure 11), with the 70-30% mix ratio showing the highest MRR values. At a depth of cut of 0.01 mm, the untreated sample showed the lowest MRR of 48 mm³/min, compared to the 70-30% mix ratio, where an MRR of 50 mm³/min showed better performance in material removal efficiency. The MRR then declined slightly for the tool with the 90-10% mix ratio sample, yielding an MRR of 49 mm³/min, which was higher than the untreated sample but lower than the 70-30% mix ratio sample – showing a higher material removal efficiency than the sample that was left untreated, but not higher than the sample with a 70-30% mix ratio. Comparable patterns were noted for cut depths of 0.007 and 0.005 mm. This demonstrates that, as shown by its high MRR values during machining, the mix ratio of 70-30% had the best material removal efficiency.

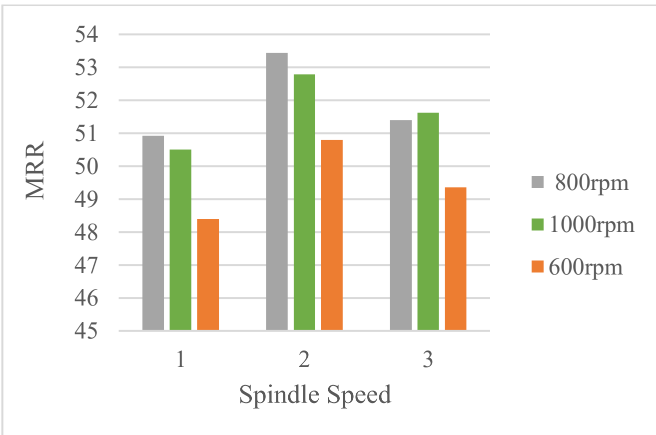

Figure 11. MRR vs. spindle speed

4. CONCLUSION AND RECOMMENDATION

The study effectively illustrated the viability and potential of creating cutting tools out of agricultural waste—more especially, carbonized cow horns and snail shells. The materials were chosen based on their special qualities, such as resistance to temperature and pressure, material properties, and chemical compatibility with other materials and reagents utilized in the experiment. Energy XRF was used to analyze the composition of the samples. The results showed that cow horns contain sizable amounts of silicon dioxide (SiO_2), ferric oxide (Fe_2O_3), calcium oxide (CaO), and sulfur trioxide (SO_3), but snail shells are primarily composed of CaO. These elements influence the final cutting tools' thermal characteristics, mechanical strength, and stiffness.

According to the findings, a blend of 70% snail shell and 30% cow horn offers a well-balanced mix of mechanical strength, thermal resistance, and efficiency, which makes it the best option for cutting tool applications. This study emphasizes the possibility of utilizing agricultural waste materials as an affordable and sustainable substitute for conventional materials used in the production of tools. This strategy adds value to otherwise discarded resources while reducing the impact on the environment through the repurposing of waste materials.

Overall, the study demonstrates that waste materials from agriculture can be efficiently converted into cutting tools that are both high-performing and useful. This creative way of repurposing waste materials supports the objectives of sustainable development and presents a viable way for the manufacturing sector to become less dependent on traditional, non-renewable resources. The results open up new avenues for investigation and refinement of other agricultural waste materials, which could expand the use and scope of environmentally friendly cutting tools in a variety of machining and manufacturing processes.

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