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Effect of storage on properties of pine needle cattle dung briquettes

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The study has been under taken to utilize abundantly available pine needles in hilly region of Uttarakhand state to mitigate the drudgery involved in collection of fuel wood from nearby forest for cooking. Pine needle briquettes have been prepared using cattle dung as binding agent in a proportion of 60:40 by weight with the help of a hydraulic press. Three levels of briquetting parameters, namely particle size (0.54, 1.5, 3.0mm), die pressure (2.8, 4.14 and 5.5MPa) and dwell time (15, 30, 45sec) have been taken. Heating value, ash content, moisture content, bulk density, crushing strength and water resistance capacity of briquettes have been evaluated. Bulk density and calorific values have decreased with increase in storage period for all types of briquettes. An overall reduction of 6.5% in bulk density and about 1.5% in calorific value has found during storage for a period of 60 days. However, all the briquettes have remained stable. Based on process optimization using RSM, briquettes prepared at highest die pressure of 5.5 MPa with 2.6 mm particle size and 15 seconds dwell time have proved to be optimal considering all the quality parameters of briquettes included in the study over the storage period of 60 days.

Keywords: Pinus roxburghii (Chi'), Pine briquettes, Die pressure, Particle size, Fuel properties

1 Introduction

Pinus roxburghii commonly known as Chir pine or long leaf Indian pine is a species of pine and is native to the Himalayas. Its home extends from Tibet and Afghanistan through Pakistan, northern states of India, Nepal and Bhutan up to Myanmar. Chir pine covers approximately 0.678 Mha area in three states of India namely Himachal Pradesh, Jammu & Kashmir and Uttarakhand. Out of these three states, Uttarakhand alone covers a major portion (0.412 Mha) of Chir pine forests. The uses of Chir pine as a timber and fuel wood are among few major indigenous uses in Uttarakhand. During autumn, the dried needles form a dense carpet on the forest floor, which the local people gather in large bundles to serve as bedding for their cattle round the year. The green needles are used to make tiny hand brooms and other decorative items. Forests with Chir pine are very prone to fire as their foliage easily catches fire, however, the pine itself is resistant to it. Except few uses, as mentioned above, this enormously available biomass from pine forests remains underutilized. In rural India, households depend on solid biomass fuel as their main energy source for cooking due to meager access to modern cooking fuels. Major portion of the fuel wood is fetched from nearby forests leading to deforestation. Cattle dung cake is another fuel source which is widely used along with fuel wood for cooking. The major fuel wood required for cooking in hilly region of the state is obtained from Chir pine forest. Considering its easy availability, ignitability and under utilization, there is a great potential of this biomass to be used as source of fuel for cooking by way of cold briquetting with cow dung as binder. The briquetting of biomass improves its handling characteristics, enhances its volumetric calorific value, reduces transportation cost and produces a uniform clean stable and environmental-friendly fuel[10]. Pandey and Dhakal (2013)[14] have recommended, pine needle as an excellent raw material with calorific value of 5230 kcal/kg for briquetting to be used in industrial boilers and kilns due to its low ash content, low moisture, relatively high carbon content, lesser smoke emission, ability to burn longer with stable and uniform temperature as compared to fuel wood. Bisht *et al.* (2014)[7] have reported calorific value of briquettes as 6501 Kcal/kg which have been produced from carbonizing pine needle (pine needle char) and adding 50 g/kg of paper to it as binder. The burning rate has been determined as 10.9 g/min with an efficiency of 39 percent. The specific fuel consumption for heating water has reported as 95 g/lit. Fixed carbon, volatile matter and ash content of the briquettes have

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*Corresponding author (E-mail: kaur.lovepreet313@gmail.com) been found as 7.1, 75.2 and 14.5 percent respectively at 4.4 percent moisture content. Muniz *et al.* (2014)[11]haveprepared charcoal from pine needle by carbonizing it at temperatures of 500,600 and 700°C and evaluated its potential use for energy. The needles have found to have ash content of 2.32% and gross calorific value of 20.30 MJ/kg. The calorific value has increased by 45% (to 29.64 MJ/kg) after carbonizing it at 600°C. This value is higher than that for charcoal made from eucalyptus (19.25 MJ/kg) and even coconut husks (23.55 MJ/kg) showing the high energy potential of pine needles. Numbers of researchers have used various other types of biomass for preparing briquettes. Wakchaure and Mani (2011)[18] have studied thermal and storage characteristics of briquettes made from mustard stalk, mixed waste of tree leaves and grasses (3:1) and wood waste using molasses, press mud and distillers dry grain as binder in 5, 10, 15 and 20 percent. The heating values have reported higher for briquettes prepared using press mud, followed by those with distiller's dry grain and molasses. Bulk density and heating value have found to decreased by 49 and 9 percent respectively during a storage period of 180 days. The briquettes produced at highest die pressure of 123.4 MPa have shown less variation in moisture content, bulk density, tumbling resistance, shattering resistance, resistance to water penetration, calorific values and ash content during this period. Okello *et al.* (2011)[13] have established the optimum conditions for converting coffee husks with molasses as binder into a densified biomass fuel by response surface methodology (RSM) using rotatable central composite experimental design (CCD). Quantity of binder, die pressure, moisture content, material particle size and dwell time have been considered as experimental parameters with response variables as density, durability and stability. Based on the results, die pressure of 14.91MPa, moisture content of 8.0% (wb) and binder content of 45.0% (wb) have been observed optimum. Under this set, the briquettes prepared have a particle density of 718.09 kg/m^3, durability of 80.77% and stability of 14.98%. Considering limited work on pine needle briquetting and its storage, the present study have been planned to study the effect of storage on properties of pine needle-cattle dung briquettes.

2 Materials and Methods

To produce pine needle-cattle dung briquettes, a manually operated perforated cylindrical die of PVC having 150 mm length and 102 mm diameter and a wooden ram designed and developed in the Research Workshop, Department of Farm Machinery and Power Engineering, College of Technology, G. B. Pant University of Agriculture and Technology, Pantnagar, Uttarakhand, India was used (Fig. 1).

The cost of the manually operated briquette making machine was ₹ 4500.00. Pine needle biomass was the raw material used for making the briquettes. Different blends of biomass and binders with water can be used. The raw materials were selected on the basis of local availability and feasibility for the average entrepreneur. The pine needles are sharp leaves occurring in the bunch, 20–35 cm long and are noticeably yellowish-brown in colour after drying. The initial moisture contents of the samples collected were in the range 23–26%. The biomass samples were

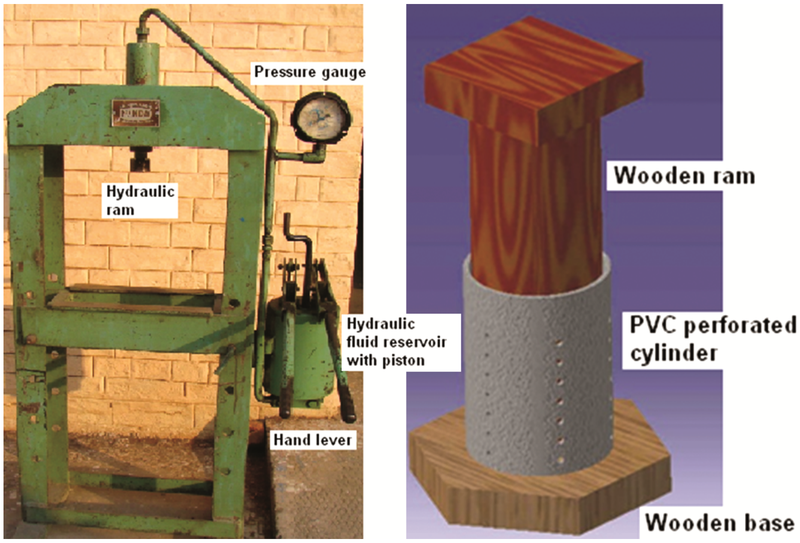

Fig. 1 — Hydraulic press and die used for making briquettes.

then dried under direct sunlight for duration of seven days. Thereafter, the average moisture content of the biomass sample was in the range 7.78–10%.

The briquette-making procedure requires additional binding material to hold the briquette together, briquette formation, storage and transportation. Binders play a significant role in the briquette production. Several types of binding materials have been used by different researchers such as organic binders, inorganic binders and compound binders. Cattle dung is an organic binder used by several briquette producers. The cattle dung binder has good binding performance and also the briquettes made using cattle dung binder show high compressive strength. The organic binders decompose easily at high temperature[9,19].

Pine needles collected from nearby pine forests were dried in sun and ground in hammer mill. Further it was sieved with Taylor's sieve and graded as fine (0.54 mm), medium (1.5 mm) and coarse (3.0 mm) particle size. It was then mixed with dried and powdered cattle dung in the ratio of 60:40 by weight. Water was added to this mixture to form dough. A fixed quantity of this was filled into a perforated cylindrical die of PVC having 150 mm length and 102 mm diameter. This sample was compressed at required pressure and dwell time using wooden ram and hydraulic press for producing briquettes at room temperature (Fig. 1). Pressure and dwell time was monitored through dial gauge of hydraulic press and a stopwatch. The briquettes were removed from die and subsequently sun-dried, were kept in plastic bags under atmospheric condition and stored for 60 days. Measurements on heating value, ash content, bulk density, moisture content, water resistance capacity and crushing strength were taken at regular intervals of 30 days.

The experiment was designed in Box-Benhken designs (BBD) of response surface methodology (RSM). In all seventeen numbers of experiments were designed with four replications each. The details of briquetting parameters and experimental combinations have been shown in Tables 1 and Table 2.

2.1 Properties of briquettes

The size of the briquettes was determined by measuring their average length and diameter. Weight of the briquettes was determined by measuring the average weight of the prepared briquettes samples using an electronic weighing balance.

The bulk density of the samples was determined using ASTM E873-82 (2013)[6]. An empty, a cylindrical-shaped container of known volume was weighed to determine its mass and then the container was filled with briquette sample and weighed again. Bulk density was estimated as

$$\text{Bulk density}(\text{kg/m}^3) = \frac{\text{Mass of the sample (kg)}}{\text{Volume of the container (m}^3)} \qquad \dots (1)$$

Water absorption test is a measure of water absorbed by the briquettes when completely immersed in water. The briquettes were immersed in water for 60 sec at room temperature[12]. The proportion of water absorbed by each briquette was calculated and the water absorption resistance determined using the formula

$$\%\ \text{Water gained by the briquette} = \frac{W_1 - W_2}{W_1} \times 100 \qquad \dots (2)$$

$$\text{Water absorption resistance} = 100 - \%\ \text{Water gained} \qquad \dots (3)$$

where, W_1 is the mass of briquette sample before the test (g) and W_2 is the mass of briquette sample after the test (g)

Table 1 — Briquetting parameters with their levels

Variables	Levels	Values
Pine needle particle size	3	0.54, 1.5, 3 mm
Die pressure	3	2.8, 4.14 and 5.5 MPa
Dwell time	3	15, 30 and 45 seconds
Replications	4	

Table 2 — Design of experiment using RSM for pine needle-cattle dung briquettes

Exp. No.	Independent variables					
	Coded levels			Actual levels		
	X_1	X_2	X_3	Particle size, mm	Die Pressure, MPa	Dwell time, S
1	-1	0	1	0.54	4.14	45
2	-1	1	0	0.54	5.5	30
3	1	0	-1	3.0	4.14	15
4	1	1	0	3.0	5.5	30
5	0	1	-1	1.5	5.5	15
6	1	-1	0	3.0	2.8	30
7	-1	0	-1	0.54	4.14	15
8	-1	-1	0	0.54	2.8	30
9	0	-1	-1	1.5	2.8	15
10	0	1	1	1.5	5.5	45
11	1	0	1	3.0	4.14	45
12	0	-1	1	1.5	2.8	45
13	0	0	0	1.5	4.14	30
14	0	0	0	1.5	4.14	30
15	0	0	0	1.5	4.14	30
16	0	0	0	1.5	4.14	30
17	0	0	0	1.5	4.14	30

The crushing strength is the ability of the briquettes to resist breaking under a compressive force. The crushing strength was measured using a texture analyzer machine. The briquette samples were placed directly on the platform of the machine plunger to be pressed. The machine applied a compressive force on the surface of briquettes until failure was encountered on them. The Crushing strength was displayed on the screen.

Moisture is defined as the amount of liquid per unit mass of the wet solid. According to the standardized test method for moisture analysis[2], 1g of the sample was retained in a hot-air-oven at 105°C for 1 h. Next, the oven-dried sample was weighed. Moisture content was estimated as

$$\text{Moisture content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \qquad \dots (4)$$

where, W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample (g) and W_3 is the weight of the empty crucible + sample after drying (g).

Volatile matter determines the percentage of the gaseous products which is released under the specific conditions of the test in the analysis of the sample free from moisture content. As per the standard test method for volatile matter analysis[4], the oven-dried sample enclosed in a crucible with a lid was placed in a muffle furnace, and maintained at 950° ± 20°C for 7 min. Therefore, the crucible was first cooled in the surrounding air and then in a desiccator, and weight loss was calculated

$$\text{Volatile matter(\%)} = \frac{W_3 - W_4}{W_2 - W_1} \times 100 \qquad \dots (5)$$

where,$W_1$is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample (g) and W_3 is the weight of the empty crucible + sample after oven-drying (g). W_4 is the weight of the empty crucible + weight after heating in muffle furnace (g).

Ash content was determined by weighing the residue remaining after burning the coal under rigidly controlled conditions of sample weight, temperature, time, atmosphere and equipment specifications. According to the standardized test method for ash content analysis [3], the remaining sample residue after volatile matter test, was heated in the muffle furnace at 700° ± 50°C for 4 h. The ash content in each sample of the briquette was estimated using the formula,

$$\text{Ash content (\%)} = \frac{W_5 - W_1}{W_2 - W_1} \times 100 \qquad \dots (6)$$

where, W_1 is the weight of the empty crucible (g), W_2 the weight of the empty crucible + sample, after volatile matter (g) and W_5 is the weight of the empty crucible + ash left in the crucible (g).

The Heating value of briquettes was determined using the bomb calorimeter, according to the standard test method for calorific value[5]. The bomb calorimeter consisted of a solid cylindrical, stainless-steel bomb inside which the combustion of fuel took place. Less than 1 g of fuel sample was placed in the crucible, a fuse wire was used to ignite the fuel and the bomb filled with oxygen gas at 25–30 atmospheric pressure. The electrode was connected to electrical supply and initial water temperature was noted. After combustion of the fuel sample, the increase in water temperature was noted for determination of calorific value. The calorific value determined by the formula

$$\text{Calorific value (kcal/kg)} = \frac{W \times \Delta \mathrm{T}}{M} \qquad \dots (7)$$

where, M is the Mass of fuel placed in the crucible (g), W is the water equivalent of the bomb calorimeter or heat capacity (cal/°C), $\Delta T = t_2 - t_1$, where t_1 is the initial temperature of water in the calorimeter (°C) and t_2 is the final temperature of water in the calorimeter (°C).

To determine the heat capacity of the bomb calorimeter, wherever nichrome fuse wire and cotton thread were used simultaneously, pure benzoic acid is used in the bomb. In this case, cotton thread was also used along with nichrome fuse wire. The heat capacity for nichrome fuse wire was 333.68 cal/g and for cotton thread it was 4180 cal/g[15]

$$W = \frac{M \times H + (E_w) + (E_t)}{\Delta T} \qquad \dots (8)$$

where, W is the water equivalent of the bomb calorimeter (2283.32 cal/°C), M the mass of the test sample, ΔT is the rise in temperature, $E_w$the correction of heat of combustion for nichrome fuse wire and E_t is the correction of heat of combustion for cotton thread

$$\mathrm{E_w} = \mathrm{M_w} \times \mathrm{H_w} \qquad \dots (9)$$

where, $\mathrm{M_w}$ is the mass of nichrome fuse wire and $\mathrm{H_w}$is the heat capacity per gram of nichrome fuse wire

$$\mathrm{E_t} = \mathrm{M_t} \times \mathrm{H_t} \qquad \dots (10)$$

where, M_t is the mass of cotton thread and H_t is the heat capacity per gram of cotton thread.

3 Results and Discussion

Figure 2 shows the pine needle-cattle dung briquettes produced with the manually operated briquettes-making perforated cylindrical die of PVC and a wooden ram. Table 3 lists the characteristic properties of briquettes.

3.1 Effect of particle size, die pressure and dwell time on ash content

Ash content of the briquettes ranged from 5.25 to 8.6 percent for all the combination of briquetting parameters (Table 3). Maximum ash content was recorded for the briquettes produced from particle size 1.5 mm, die pressure 4.14 MPa and dwell time 30 seconds where as it was found minimum for the briquettes produced from 1.5 mm particle size, 5.5 MPa die pressure and 15 seconds dwell time. Statistically the effect of dwell time on ash content was found to be highly significant followed by the effect of particle size at 1% level of significance ($p<0.01$). The effect of die pressure on ash was also found to be significant at 10% level of significance. The increase in ash content with the reduction in particle size (Fig. 3(a)) is due to the fact that smaller particle sizes get compacted easily leaving fewer pore spaces between them and hence leading to incomplete combustion. Similar results have been reported by Tokan *et al.*[17]. The effect of die pressure on ash content (Fig. 3(b)) was less pronounced. Wakchaure and Mani (2011)[18]have also reported non-significant effect of die pressure on ash content. The ash content was found to have positive correlation with dwell time (Fig. 3(c)). Higher dwell time resulted in more compaction of briquettes resulting in fewer pore space and incomplete burning and hence more ash content. The following regression equation was established between briquetting parameters and ash content with r_2 as 0.944.

Fig. 2 — Briquettes prepared with cattle dung and pine needle.

Table 3 — Characteristic properties of briquettes

Experiment no.	Dependent variable					
	Ash content, %	Heating value, kJ/kg	Moisture content, %	Crushing strength, kg	Bulk d ensity, g/cc	Water resistance capacity,%
1	8	10185.62	3.72	65.8	0.227	97.26
2	8.2	10157.71	4.05	44.01	0.232	97.66
3	6.6	10377.11	3.82	71.84	0.167	97.14
4	6.15	10424.23	3.02	57.73	0.182	96.82
5	5.25	10508.92	3.22	47.74	0.201	97.15
6	7.84	10208.29	4.42	60.4	0.17	97.35
7	7.65	10222.09	2.95	50.01	0.215	97.15
8	8.05	10180.91	3.85	62.39	0.223	97.34
9	7.3	10279.98	4.62	91.14	0.181	97.48
10	7.75	10228.17	4.57	65.22	0.198	97.59
11	6.5	10382.50	4.45	65.33	0.164	97.3
12	6.59	10341.76	5.2	48.09	0.221	97.79
13	8.6	10150.52	3.7	43.25	0.211	98.02
14	8.15	10150.11	4.05	46.22	0.217	98.14
15	8.2	10174.84	3.83	38.34	0.211	98.12
16	7.94	10197.10	3.52	44.1	0.212	98.09
17	7.8	10222.78	3.77	40.01	0.217	97.84
p- value	0.0013	0.0009	0.0254	0.0024	0.0002	0.0034
f-value	13.23	14.69	4.79	10.82	25.27	9.65
r^2	0.944	0.949	0.860	0.9329	0.9701	0.9254
Lack of fit (LOF)	NS	NS	NS	NS	NS	NS

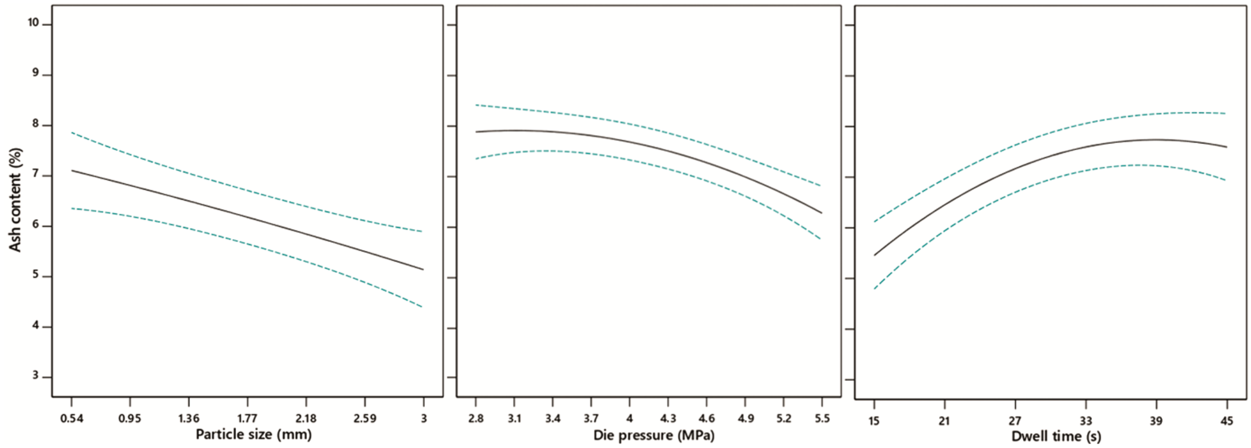

Fig. 3 — (a) Effect of particle size on ash content, (b) effect of die pressure on ash content, and (c) effect of dwell time on ash content.

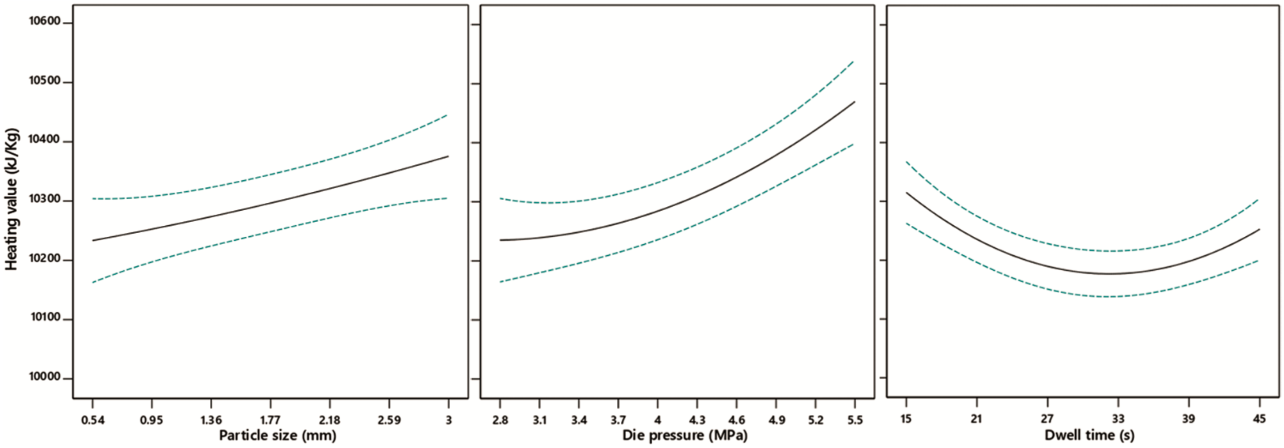

Fig. 4 — (a) Effect of particle size on heating value, (b) effect of pressure on heating value, and (c) effect of dwell time on heating value.

$$Y = 8.14 - 0.60X_1 - 0.30X_2 + 0.26X_3 - 0.46X_1X_2 + 0.80\,X_2X_3 - 0.52X_2^2 - 0.89X_3^2 \quad \ldots (11)$$

where, Y is total ash content (%) and X_1, X_2 and X_3 denotes particle size, die pressure and dwell time, respectively.

3.2 Effect of particle size, die pressure and dwell time on heating value

Heating value was observed maximum (10508.92 kJ/kg) for briquettes produced from 1.5 mm particle size at 5.5MPa die pressure and for 15 seconds dwell time whereas it was observed minimum (10150.11 kJ/kg) for briquettes produced from 1.5 mm particle size, 4.14MPa die pressure and 30 seconds dwell time. All the briquetting parameters were found highly significant ($p<0.01$). As per statistical analysis, the dwell time was found to be highly significant followed by die pressure and particle size at 1% significance level ($p<0.01$). Particle size and die pressure were found to be having positive correlation (Fig. 4 (a and b)) which meant that heating value would increase with the increase in value of these two parameters. However, dwell time (Fig. 4(c)) was observed to have negative correlation with heating value. The explanation for this is that larger particle size briquettes have greater number of pore spaces allowing oxygen to flow easily through it for complete combustion and hence more heating value. Similar results have been reported by Abdullahi *et al.* (2011)[1] and Tokan *et al.* (2014)[17]. Higher die pressure would help in packing more briquetting material per unit volume and hence more heating value was obtained. Wakchaure and Mani (2011)[18] have also reported increase in calorific value of briquettes with increase in die pressure. Regression equation, as given under, with r_2 = 0.9497 was established between briquetting parameters and heating value.

$$Y = 10179.07 + 80.71X_1 + 38.51X_2 - 31.25X_3 + 59.76X_1X_2 - 85.63X_2X_3 + 55.79X_2^2 + 104.84X_3^2 \quad \ldots (12)$$

where, Y is total ash content (%) and X_1, X_2 and X_3 denotes particle size, die pressure and dwell time,

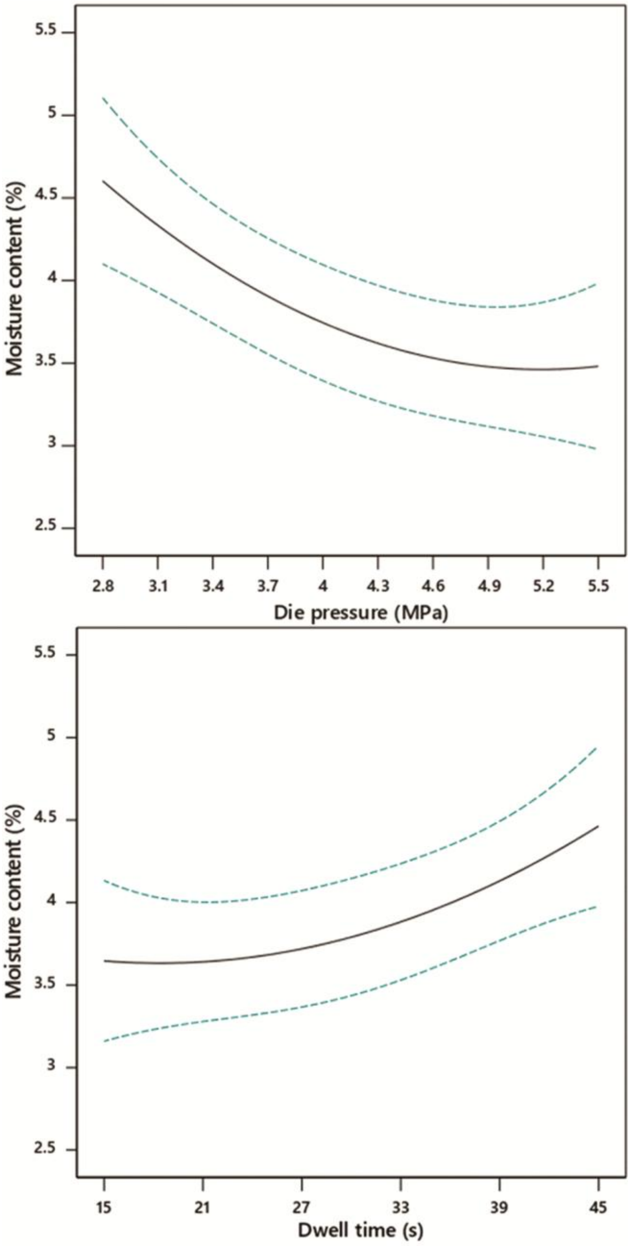

Fig. 5 — (a) Effect of pressure on moisture content, and (b) effect of dwell time on moisture content.

respectively.

3.3 Effect of particle size, die pressure and dwell time on moisture content

The moisture content was observed high for the briquettes produced with particle size of 1.5 mm, die pressure 2.8 MPa and dwell time of 45 seconds while it was low for particle size of 0.54 mm, die pressure 4.14 MPa and 15 seconds dwell time. Statistical analysis revealed that all the three briquetting parameters had highly significant effect ($p<0.01$) on moisture content of the briquettes. Among all the three parameters, effect of die pressure was found to be highly significant followed by dwell time and particle size at $p<0.01$. However, the model was found significant at 5% level of significance with p-value as 0.0254. LOF was again observed insignificant with r_2 -value for the model as 0.860. Increase in die pressure resulted in lower moisture content and vice versa which may be due to draining of excess available moisture (Fig. 5(a)). The higher moisture content with smaller particle size could be due to more surface area available for retention/adhesion of moisture. Higher dwell time might have helped in expelling out the excess moisture(Fig. 5(b)) from the briquettes. Tokan *et al.* (2014)[17]also reported the range of moisture content from 10 to15 percent for best performance. The moisture content of the briquettes was observed in the range of 2.95 to 5.20 percent which is much below the suggested value.

3.4 Effect of particle size, die pressure and dwell time on crushing strength

Crushing strength of the briquette is one of the important parameters used to assess its ability to be handled, packed and transported without breaking. The briquettes produced from 1.5mm particle size, 2.8 MPa die pressure and 15 seconds dwell time had maximum crushing strength of 91.14 kg whereas it was minimum (38.34 kg) for briquettes produced from 1.5mm particle size, 4.14MPa die pressure and 30 seconds dwell time. The effect of all the operating parameters was observed to be highly significant ($p<0.01$).Among the operating parameters, dwell time was observed to have highly significant effect followed by die pressure and particle size respectively on the basis of their F-values. The regression model was also found to be highly significant ($p<0.01$) with p-value as 0.0024, r_2 -value 0.9329 and insignificant LOF respectively. The smaller particle size at low value of dwell time indicated higher strength but decreased with the increase in dwell time (Fig. 6(a)). Higher crushing strength was observed with higher values of die pressure and dwell time (Fig.6(b)). This may be due to better compression of briquette material. However, for lower dwell time and die pressure the crushing strength was again found to be higher. Stephen *et al.* (2013)[16] reported that the major contributing factors to the bond formed during densification of briquettes may be the mechanical interlock of the fibers and adhesive force between the particles. Smaller particle size might have lead to better adhesion between the particles and hence higher crushing strength was obtained. It was also reported that higher compacting pressure increased binding force and adhesion between the particles resulting into increased compressive strength.

3.5 Effect of particle size, die pressure and dwell time on bulk density

The bulk density of briquette was found to be in the range of 0.164 to 0.232g/cc with maximum for briquettes produced with particle size 0.54 mm, die pressure 5.5MPa and 30 seconds dwell time while it was minimum for the briquettes produced from 3.0 mm particle size, 4.14MPa die pressure and 45 seconds dwell time. The effect of all the three parameters on bulk density was found to be highly significant ($p<0.01$). However, the effect of particle size was observed to be more pronounced followed by dwell time and die pressure. A linear relation was observed to exist between the bulk density and operating parameters with r_2 -value as 0.9701, p-value as 0.0002 and LOF as non-significant. The briquettes produced from smaller particle size as well as with more dwell time had higher bulk density and vice-versa (Fig. 7(a)). This is due to the fact that briquette material with smaller particle size could be compacted more in comparison to material with larger particle size resulting into more weight per unit volume and hence higher bulk density. Also,

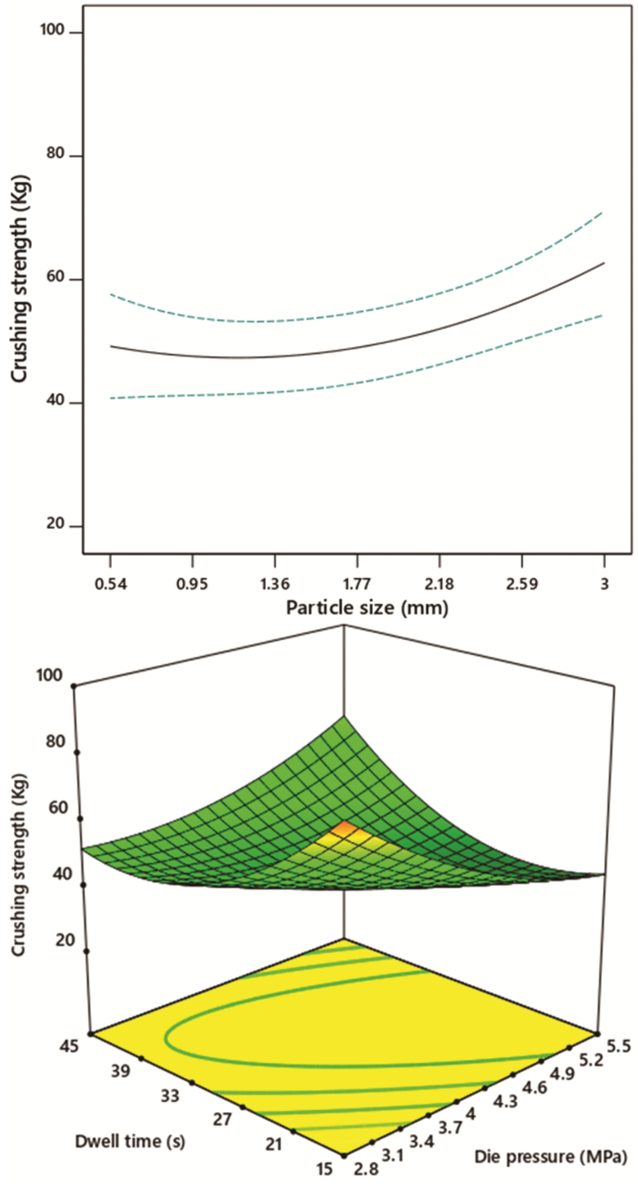

Fig. 6 — (a) Effect of particle size on crushing strength, and (b) effect of pressure on crushing strength.

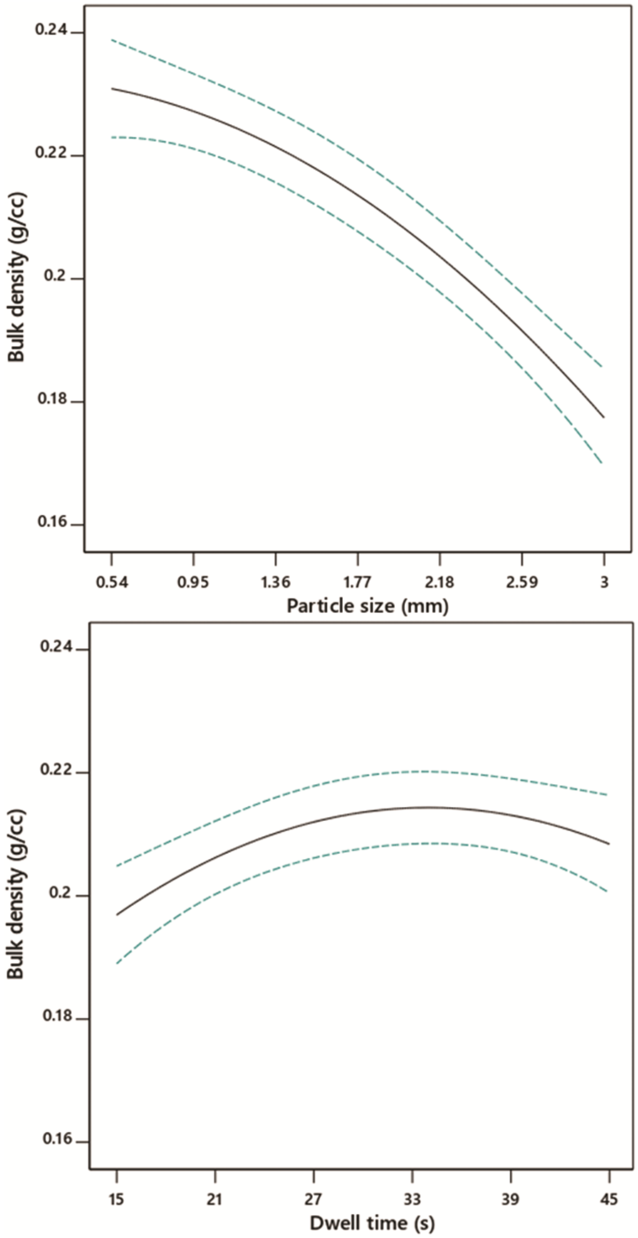

Fig.7 — (a) Effect of particle size on bulk density, and (b) effect dwell time on bulk density.

increase in dwell time will result into higher bulk density due to better packing of the briquetting material (Fig. 7(b)). Also, with higher die pressure more material per unit volume can be compressed resulting into higher bulk density.

3.6 Effect of particle size, die pressure and dwell time on water resistance capacity

The result of water resistance capacity (WRC) of the briquettes revealed that it varied from 96.82 to 98.14% being minimum for the briquettes produced from 3.0 mm particle size at 5.5 MPa die pressure and 30 seconds dwell time. Highest WRC was observed for the briquettes produced from 1.5 mm particle size, 4.14 MPa die pressure and 30 seconds dwell time. Statistical analysis revealed that all the briquetting parameters had highly significant effect on WRC ($p<0.01$). Further statistical analysis revealed that among the operating parameters, die pressure was observed to be highly significant parameter followed by dwell time and die pressure at 1% level of significance. The regression model was also found to be highly significant with r_2 -value as 0.9254 (p-value 0.0034) and non-significant LOF. WRC increased slightly with the increase in die pressure and reduction in particle size (Fig. 8 (a and b)). This may be due to the fact that higher die pressure and smaller particle size decreased the water absorption capacity by sealing the voids between the particles preventing water infiltration and passage. Also higher compaction pressure might have brought the smaller particles closer preventing water absorption and hence higher WRC. Similar results have been reported by Davies and Davies (2013)[8]. Dwell time was also found to have positive correlation with WRC indicating increase in its value with the increase in dwell time.

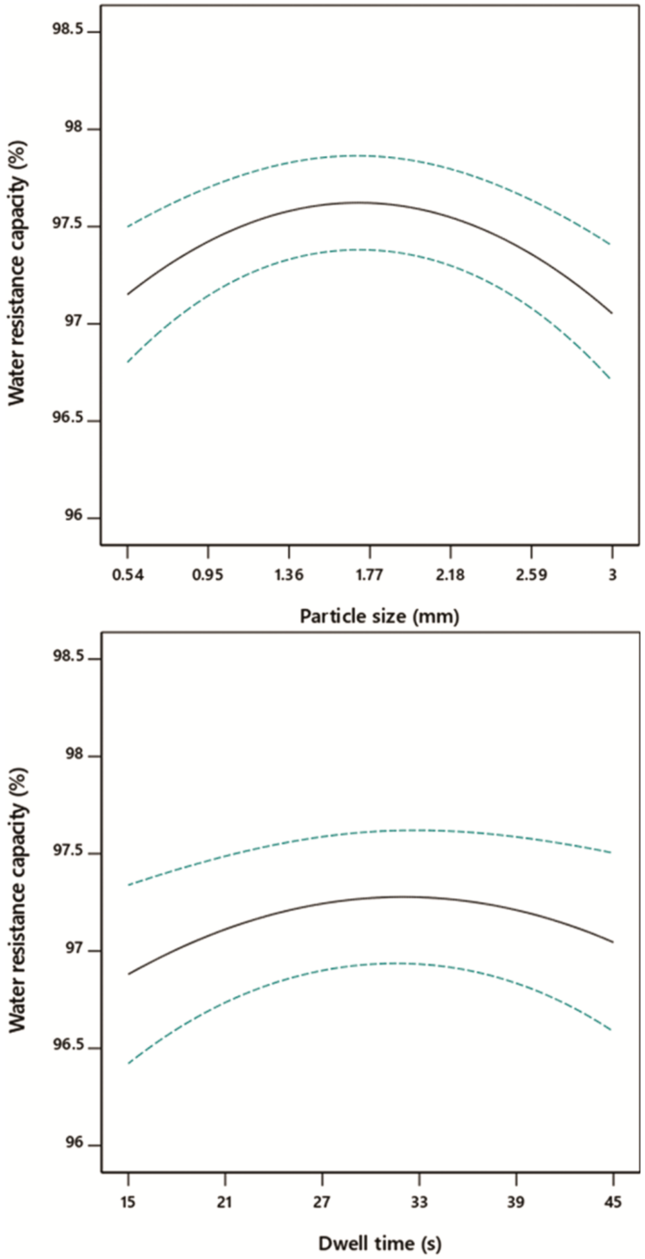

Fig. 8 — (a) Effect of particle size on water resistance capacity, and (b) effect of dwell time on water resistance capacity.

3.7 Effect of Storage Period on Quality of Briquettes

3.7.1 Ash content

It was observed that ash content of briquettes increased with increase in storage period for all types of briquettes, Table 4(a). Percent increase in ash content of the briquettes varied from 21.9 to 45.4 over the time of 60 days. With increase of storage time, ash content increased resulting in decrease in calorific values of biomass briquettes. It might be due to biological degradation and/or evaporation of volatile matter from the biomass briquettes. The statistical analysis showed that ash content was significantly influenced by the study variables at 5% level of significance.

3.7.2 Calorific value

Calorific value of briquettes decreased with increase in storage time. The briquettes prepared at highest die pressure and fine particle size showed less variation in calorific values, Table 4(a). Percent decrease in calorific values of the briquettes varied from 1.54 to 1.59 over the time of 60 days. The percent of decrease in calorific value was lowest for briquettes prepared at 5.5 MPa die pressure with 15s dwell time and 1.5mm particle size. The results were statistically significant at 5% level of significance.

3.7.3 Moisture content

With increase in storage period, moisture content of briquettes made of all biomass materials and binders at

Table 4 — (a) Effect of storage period on ash content and Heating value of biomass briquette

Briquette storage characteristic	Ash content,%			Heating value, kJ/kg		
Period	0	30	60	0	30	60
1	8	10.2	10.9	10185.62	10106.62	10023.62
2	8.2	10.45	11.15	10157.71	10078.71	9995.71
3	6.6	8.9	9.6	10377.11	10298.11	10215.11
4	6.15	7	7.7	10424.23	10345.23	10262.23
5	5.25	5.89	6.59	10508.92	10429.92	10346.92
6	7.84	8.9	9.6	10208.29	10129.29	10046.29
7	7.65	9.7	10.4	10222.09	10143.09	10060.09
8	8.05	10.4	11.1	10180.91	10101.91	10018.91
9	7.3	9.3	10	10279.98	10200.98	10117.98
10	7.75	8.75	9.45	10228.17	10149.17	10066.17
11	6.5	7.25	7.95	10382.50	10303.50	10220.50
12	6.59	8.3	9	10341.76	10262.76	10179.76
13	8.6	10.05	10.75	10150.52	10071.52	9988.52
14	8.15	10.5	11.2	10150.11	10071.11	9988.11
15	8.2	9.75	10.45	10174.84	10095.84	10012.84
16	7.94	10.05	10.75	10197.10	10118.10	10035.10
17	7.8	9.45	10.15	10222.78	10143.78	10060.78

Table 4 — (b) Effect of storage period on moisture content and crushing strength of biomass briquette

Briquette storage characteristic	Moisture content %			Crushing Strength Kg		
Period	0	30	60	0	30	60
1	3.72	3.22	2.77	65.8	64.8	63.85
2	4.05	3.85	3.68	44.01	43.01	42.06
3	3.82	3.32	2.87	71.84	70.84	69.89
4	3.02	2.92	2.8	57.73	56.73	55.78
5	3.22	3.05	2.98	47.74	46.74	45.79
6	4.42	3.92	3.47	60.4	59.4	58.45
7	2.95	2.45	2	50.01	49.01	48.06
8	3.85	3.35	2.9	62.39	61.39	60.44
9	4.62	4.12	3.67	91.14	90.14	89.19
10	4.57	4.41	4.28	65.22	64.22	63.27
11	4.45	3.95	3.5	65.33	64.33	63.38
12	5.2	4.7	4.25	48.09	47.09	46.14
13	3.7	3.2	2.75	43.25	42.25	41.3
14	4.05	3.55	3.1	46.22	45.22	44.27
15	3.83	3.33	2.88	38.34	37.34	36.39
16	3.52	3.02	2.57	44.1	43.1	42.15
17	3.77	3.27	2.82	40.01	39.01	38.06

Table 4 — (c) Effect of storage period on bulk density and water resistance capacity of briquettes

Briquette storage characteristic	Bulk density g/cc			water resistance capacity %		
Period	0	30	60	0	30	60
1	0.227	0.219	0.212	97.26	97.76	98.16
2	0.232	0.224	0.217	97.66	98.16	98.56
3	0.167	0.159	0.152	97.14	97.64	98.04
4	0.182	0.174	0.167	96.82	97.32	97.72
5	0.201	0.189	0.184	97.15	97.65	98.05
6	0.17	0.162	0.155	97.35	97.85	98.25
7	0.215	0.207	0.2	97.15	97.65	98.05
8	0.223	0.215	0.208	97.34	97.84	98.24
9	0.181	0.173	0.166	97.48	97.98	98.38
10	0.198	0.19	0.183	97.59	98.09	98.49
11	0.164	0.156	0.149	97.3	97.8	98.2
12	0.221	0.213	0.206	97.79	98.29	98.69
13	0.211	0.203	0.196	98.02	98.52	98.92
14	0.217	0.209	0.202	98.14	98.32	98.72
15	0.211	0.203	0.196	98.12	98.41	98.81
16	0.212	0.204	0.197	98.09	98.59	98.99
17	0.217	0.209	0.202	97.84	98.34	98.74

selected die pressures decreased, Table 4(b). The briquettes prepared at the highest die pressure showed less variation in moisture content during storage period, since the initial moisture content of such briquettes was low. Statistical analysis showed that die pressure, materials and binders affected the briquette quality significantly at 5% level of significance during the storage period. The decrease in moisture content with increase in storage period might be due to the fact that the briquettes were stored in summer season. Briquettes being hygroscopic in nature de-absorb moisture when ambient temperature and humidity facilitated the same. Absorption of moisture is a negative factor and need to be avoided, as it adversely affected physical and thermal properties.

3.7.4 Crushing strength

Crushing strength, one of the important characteristics during transportation and storage of briquettes, decreased with increase in storage time for briquettes made from pine needle and cattle dung at three die pressures, dwell time and particle size (Table 4(b)). The largest reduction in crushing strength of 5% with increase in storage period for briquettes obtained at die pressure of 4.14 MPa, particle size of 1.5mm and 30s of dwell time. In general, briquettes prepared at lowest die pressure (2.8MPa) showed less variation in Crushing strength during the storage period. Thus, briquettes made at lowest die pressure were superior as compared to briquettes made at other two die pressures (4.14,5.5 MPa) in terms of shattering resistance. This might be due to the fact that press mud had higher binding and Crushing strength. The variation in Crushing strength of briquettes made of different particle size and dwell time at different die pressures was significant with storage time at 5% level of significance.

3.7.5 Bulk density

Bulk density decreased with increase in storage time for all types of briquettes, Table 4(c). In general, the briquettes prepared at highest die pressure and fine particle size showed less variation in bulk density over the storage period. Briquettes, made at die pressure of 5.5MPa, 0.54mm particle size and 30s dwell time , exhibited up to 6.4% variation in bulk density over storage period of 60 days, and up to 9.14% variation observed for briquettes prepared at die pressure of 4.14MPa, 3.0mm particle size and 45s dwell time. The variation in bulk density of briquettes with storage time was significant at 5% level of significance. An optimal level of moisture content and bulk density for different types of briquettes is desirable.

3.7.6 Water resistance capacity

The resistance to water penetration of briquettes increased (0.59 to 0.93%) with increase in storage time of the briquettes, at all three die pressures, particle size and dwell time (Table 4(c)). The largest increase of 0.93% in resistance to water penetration was observed for briquettes made at die pressure of 5.5MPa, 3mm particle size and 30s dwell time during the storage period. In general, briquettes prepared with medium particle size (1.5mm) and pressure (4.14MPa) showed less variation in resistance to water penetration over the storage period.

4 Conclusion

From the present study it has been concluded that the pine needle and cattle dung briquettes (60% pine needle and 40% cattle dung) produced from 2.6 mm particle size at 5.5 MPa die pressure and 15 seconds dwell time shows best result considering all the quality parameters included in the study over the storage period of 60 days. The ash content and heating value have been found as 4.6 percent and 10593.17 kJ/kg respectively.

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