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Article

A Completed Proved of Rapid Nondestructive Prediction Model of Wood Chip Biomass Higher Heating Value Ready for Industrial Updating

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Abstract: Nepal, primarily an agricultural country, heavily relies on agricultural residue and fuelwood for daily energy requirements. In 2022, total energy consumption was 640 PJ, with traditional sources accounting for 64.17%, and fuelwood comprising 58.53% of total fuel consumption. The estimated potential supply of agricultural residue is 26 million tonnes, yielding about 442 million GJ of energy in 2021. Biomass trading often emphasizes volume or weight, necessitating a rapid and non-destructive assessment of energy properties for mutual benefit, aiding in the identification, management, and utilization of biomass sources. In this study, 200 biomass samples were collected in two batches (126 and 74 samples) from various locations in Nepal. Using Partial Least Squares Regression (PLSR), a model was developed correlating higher heating value (HHV) from a bomb calorimeter and spectral data from Fourier Transform near-infrared spectroscopy (FT-NIRS) (3595 – 12,489 cm⁻¹) sensor. PLSR models incorporated raw spectra, eight preprocessing techniques, the multi-preprocessing five-range method, and a genetic algorithm. Outliers in the first batch were identified, and the first batch divided into an 80% calibration set and a 20% validation set, while the second batch was designated as an unknown sample set. The optimum PLSR model, utilizing first derivative preprocessing, improved accuracy by 6.77%, with coefficients of determination in the calibration set, validation set, and unknown set as 0.9694, 0.9578, and 0.8089, respectively. Root mean square errors were 132.4790 J/g, 189.4800 J/g, and 360.8845 J/g for the calibration set (RMSEC), validation set (RMSEP), and unknown set (RMSEUN), respectively. The prediction to deviation ratio (RPD) for the validation set and unknown set was 4.9 and 2.4, respectively. The cross validation model of combined sample data of every sets showed the R²_{cv} of 0.95 and RPD_{cv} of 4.6 indicating the model could serve as a reliable and swift non-destructive alternative for evaluating biomass HHV using NIRS and ready for updating for biomass industrial use. However, incorporating a larger number of representative samples is crucial to enhance accuracy and develop a more comprehensive global model for predicting biomass HHV.

Keywords: biomass energy; higher heating value; near-infrared spectroscopy; partial least squares regression

1. Introduction

Globally, biomass serves as a significant energy source, with over 90% of biomass energy generated through direct combustion methods such as wood-fired heating plants and wood pellet burners [1]. The global energy-related CO₂ emissions rose to 33 billion metric tons in 2021 from 20.5 billion metric tons in 1990, highlighting the urgent need for the world to shift towards cleaner renewable energy sources [2]. In 2020, the total primary energy supply was 585 EJ globally, with fossil fuels accounting for 80% (coal 27%, oil 29%, and gas 24%), nuclear power contributing 29.2 EJ, 5%, and renewable energy technologies such as solar, wind, hydro, biomass, and geothermal accounting for 15%. According to International Energy Agency - Bioenergy, especially modern biofuels account for the largest renewable energy source constituting 55% of renewable energy and over 6% of the global energy supply [3]. Despite fossil fuels currently dominating the global energy supply, there has been a paradigm shift towards promoting and using renewable energy technologies, playing a pivotal role in mitigating climate change, improving human health, and decarbonizing energy systems [4].

Nepal, primarily an agricultural country, heavily relies on traditional biomass [5], with 77% of the country's energy consumption coming from sources like cattle dung, wood fuel, and agricultural residues [6]. The estimated supply potential of agricultural residues in Nepal was 26 million tons in 2021, producing about 442 million GJ of energy, highlighting the potential for transitioning towards modern biofuels. In 2022, Nepal's total energy consumption reached 640PJ, with traditional sources accounting for 64.17% and fuelwood dominating at 58.53% of total fuel consumption. With abundant agricultural residues and significant forest cover encompassing 45% of its territory, Nepal is well-positioned for the production and utilization of modern biofuels [7]. However, properly identifying biomass in terms of its energy properties, managing it efficiently, and utilizing it effectively poses significant challenges. Therefore, research on characterizing Nepal's biomass for rapid and reliable evaluation of its energy properties non-destructively is of utmost importance and constitutes a national priority research domain.

Near Infrared Spectroscopy (NIRS) has broad multidisciplinary applications in quality control, quality assurance, and real-time screening across various fields, including energy, agriculture, agri-food, medicine, polymer, chemical production, cosmetics, and water quality analysis [8-10]. NIRS utilizes the near-infrared light spectrum to analyze molecular information in samples of various states, shapes, and thicknesses by measuring absorption bands resulting from overtones and combination excitation [11], making it valuable for assessing compounds and their concentrations. It offers a wide range of benefits over conventional destructive techniques as it provides accurate qualitative and quantitative results in under a minute non-destructively, without using any chemicals. This makes it an eco-friendly and non-contact analysis technique.

Biomass effectively absorbs near infrared radiation within the NIR range of 3598 to 12489 cm⁻¹ [12, 13]. Therefore, in the energy sector, the combination of NIRS with chemometrics is particularly useful for predicting and evaluating biomass energy properties, including HHV, proximate analysis parameters (moisture content, volatile matter, fixed carbon, ash content), and ultimate analysis parameters (carbon, hydrogen, sulfur, oxygen, and nitrogen). These parameters are vital components in the design and analysis of any bioenergy system [14]. Shrestha, *et al.* studied the effect of combined non-wood and wood spectra of biomass chips for the rapid prediction of ultimate analysis parameters using FT-NIRS [15]. Elena, *et al.* monitored the qualitative properties of commercialized pellets to rapidly assess whether they were made of virgin or chemically treated wood using FT-NIRS [16]. Fumin, *et al.* developed a model combining partial least squares and artificial neural networks to estimate sugarcane stalk bending strength and flexural rigidity via FT-NIR spectrum calibration [17]. Cristiano, *et al.* predicted the lignin content of tropical Amazon woods using FT-NIRS [18]. Livia, *et al.* studied the potential of FT-NIRS for predicting cellulose nanofibril quality in commercial bleached kraft pulp of Eucalyptus [19]. Shrestha, *et al.* developed the PLSR model combined with chip and ground biomass FT-NIR spectra for the evaluation of proximate analysis parameters as an alternative to a thermogravimetric analyzer [20]. Phoomwarin, *et al.* enhanced the evaluation of sugarcane energy content for energy cane varieties selection purposes in breeding programs using FT-NIRS [21].

Shrestha, *et al.* conducted a comprehensive assessment of biomass properties for energy usage using FT-NIRS and spectral multi-preprocessing techniques [12]. All the research mentioned above highlights the potential of FT-NIRS for the rapid and non-destructive evaluation of different biomass properties. Therefore, in this study, we have proved a partial least square regression (PLSR) based model developed for the rapid non-destructive prediction of the HHV of biomass for energy usage in industry.

The research output of this study will be applicable to various stakeholders. This includes biomass traders who trade biomass based on volume or weight rather than its actual energy content, engineers and researchers involved in the design and development of different bio-based technologies, particularly in combustion via combined heat and power systems, industries reliant on biomass for the production of quality biomass pellets and briquettes, and policymakers seeking accurate forecasts of biomass energy, mainly from fast-growing trees and agricultural residues.

2. Materials and Methods

Figure 1 illustrates the overall research methodology for the rapid and non-destructive evaluation of the HHV of biomass collected from various locations in Nepal, employing a combination of NIRS with PLSR-based models.

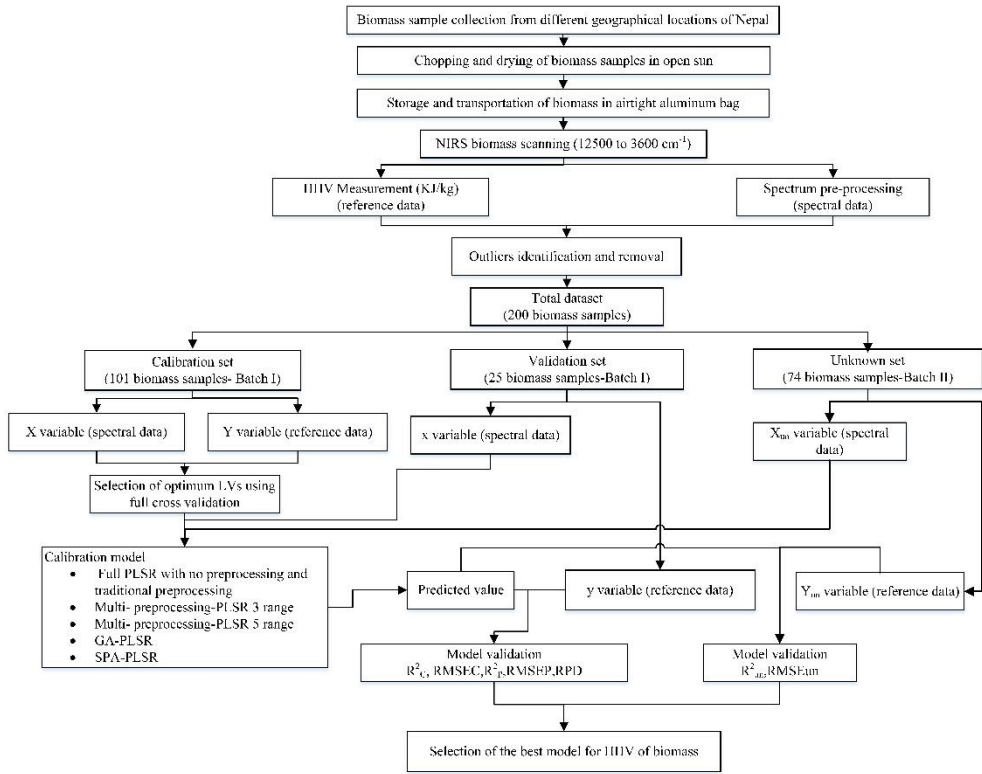


Figure 1. Overall research methodology for a rapid and nondestructive prediction of biomass HHV using FT-NIRS and PLSR.

2.1. Sample Collection

Figure 2 shows the geographical location of Nepal, district-wise, from which biomass samples were collected. The biomass samples include five varieties each of fast-growing trees: *Alnus nepalensis*, *Pinus roxburghii*, *Bambusa vulgaris*, *Bombax ceiba*, and *Eucalyptus camaldulensis* and of agricultural residues: *Zea mays* (cob), *Zea mays* (shell), *Zea mays* (stover), *Oryza sativa*, and *Saccharum officinarum*. The biomass samples were collected in two batches. In the first batch (I) a total of 126 biomass samples were collected in January to February 2021, which were used for developing the calibration model. The second batch (II) includes 74 biomass samples that were collected in January 2022, which were used as an unknown sample set to prove the performance of the model to be used in biomass industries. All the samples were manually chopped into pieces smaller than 30 mm by 15

mm, and then dried in the open sun. They were stored in airtight aluminum bags to prevent the exchange of air and moisture. The bags were only opened during FT-NIRS scanning of specific biomass.



Figure 2. Geographic location of Nepal, from which fast-growing trees and agricultural residues biomass samples have been collected.

2.2. FT-NIRS Sample Scanning

Biomass samples from both batches (I and II) were scanned across the full NIR wavenumber range (3598–12489 cm^{-1}) using an FT-NIR spectrometer (MPA, Bruker, Ettlingen, Germany) in diffuse reflectance with a sphere macro sample rotating setup, while maintaining consistent laboratory conditions, i.e., air conditioning at a temperature of $25\pm2^\circ\text{C}$ [15]. Figure 3 displays the raw spectra obtained after scanning biomass samples of fast-growing trees and agricultural residues used in this research. The biomass located at the bottom of the scanning cup was collected and immediately subjected to the evaluation of HHV using a bomb calorimeter.

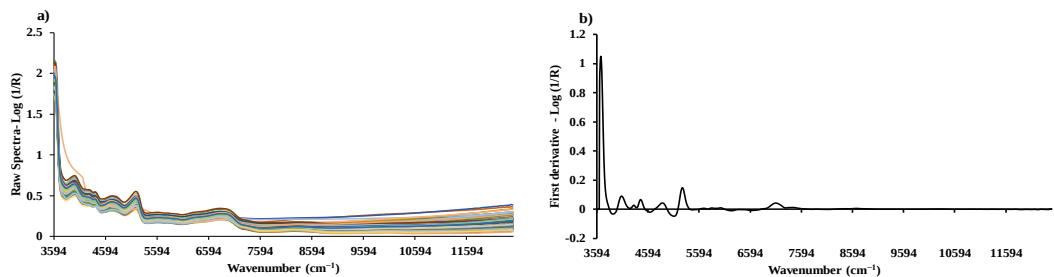


Figure 3. (a) Raw spectra, and (b) preprocessing of raw spectra using first derivative (average), obtained from FT-NIRS scanning within the range of 3598–12489 cm^{-1} .

2.3. Higher Heating Value (Reference Data)

Approximately 0.5 ± 0.2 g of biomass was collected from the bottom of the FT-NIR spectrometer scanning cup. The biomass was manually compressed using a laboratory-scale pelletizer to form biomass tablets, which were then weighted using an electronic balance (Mettler Toledo JS1203C). The

HHV was determined using an automatic bomb calorimeter (IKA C 200, Staufen, Baden-Württemberg, Germany) employing the isoperibol method [12].

2.4. Spectral Preprocessing

The raw spectra obtained from the FT-NIR spectrometer mainly contains baseline shifts and noise, attributed to instrument artifacts, light scattering, and variations in moisture and temperature. Therefore, in this study, the raw spectra were preprocessed using ten different techniques, including constant offset, SNV, MSC, first derivative, second derivative, vector normalization, min-max normalization, mean centering, first derivative + vector normalization, and first derivative + MSC. Additionally, the raw spectra were pretreated using multi-preprocessing techniques with 3-range and 5-range methods proposed by Shrestha et al [12, 15]. Following the preprocessing of the raw spectra, PLSR-based models were developed.

2.5. PLSR Based Model Development

Before model development, outliers from the reference data were identified using equation (1).

$$\frac{(X_i - \bar{X})}{SD} \geq |\pm 3| \quad (1)$$

Where, X_i represents the measured value of sample i , while $\bar{X}$ and SD denote the average and standard deviation, respectively, of the measured values across all samples [12, 15, 20]

If equation (1) is satisfied, the sample is considered an outlier, and both the reference value and spectral data of that specific sample are excluded from the dataset.

Based on the available dataset, after removing outliers, the total dataset is divided into a calibration set (80%) and a validation set (20%) manually where the calibration set comprises the maximum and minimum HHV values of biomass samples to ensure the model's capability in predictions of prediction set. Full-PLSR models were developed using raw spectra and ten different preprocessing methods. Similarly, SPA-PLSR and GA-PLSR selected NIR bands based models were also developed using raw spectra and ten different preprocessing methods. Additionally, PLSR models based on multi-preprocessing 3-range and 5-range techniques were developed. The performance of the models was evaluated based on R^2_c , RMSEC, R^2_p , RMSEP, RPD, and bias values. The best-performing models were selected based on a comparison between higher R^2_c , R^2_p , and RPD values and lower RMSEC and RMSEP values. Furthermore, best performing models were validated using the unknown sample set. The performance of the model based on unknown sample set was proved in terms of R^2_{un} , RMSEUN, and RPD_{un}.

Then updating model was developed by combining the samples in calibration set, validation set and unknown set and made the model using the best pretreated method spectra and the best PLSR algorithm selected in previous procedure. The updating model was validated by leave one out cross validation. The performance of the updating model based on cross validation was reported in terms of R^2_{cv} , RMSECV, and RPD_{cv}.

The interpretation of R^2 in the calibration set, validation set, and unknown sample set is conducted according to the guidelines established by Williams, *et al.* [22], while RPD values for the respective sets were assessed based on the guidelines provided by Zornoza, *et al.* [23].

All PLSR based modeling was carried out using the built-in MATLAB R2020b code (MathWorks, USA).

3. Results

Table 1 shows the descriptive statistics of samples in calibration set, validation set and unknown set.

Table 1. Descriptive statistics of the HHV of the biomass samples in calibration set, validation set and unknown set used in PLSR based model development.

Statistic	Calibration set	Validation set	Unknown Set
Number of sample	97	26	74
Maximum (kJ/kg)	19403	18562	18616
Minimum (kJ/kg)	14965	15130	15031
Mean (kJ/kg)	17045	17108	16944
Standard deviation (kJ/kg)	788	854	875

As explained in section 2.5, outliers for reference data were calculated using equation (1) before model development. Out of the 126 biomass samples collected from the first batch, 4 samples were identified as outliers and subsequently removed from the total dataset to develop PLSR-based models for evaluating the HHV. Table 2 shows best performing models obtained from Full-PLSR, GA-PLSR, SPA-PLSR, MP-PLSR: 3 range, and MP-PLSR: 5 range methods. Out of the five different PLSR-based models, Full-PLSR with the first derivative, having a segment and gap of 5 each, and number of latent variable (LVs) of 15 demonstrates the best performance with an R^2_c of 0.9694, RMSEC of 132.4790 kJ/kg, R^2_p of 0.9578, RMSEP of 186.0301 kJ/kg, RPD of 4.8759 and bias of 9.2028 kJ/kg. The accuracy of Full-PLSR improved by 6.77% with spectral preprocessing using the first derivative compared to that of Full-PLSR using raw spectra.

Table 2. Results of the PLSR-based model for the HHV (kJ/kg) of biomass, with best performing model highlighted in bold.

Algorithm	Preprocessing	LVs	Calibration Set		Validation set			
			R^2_c	RMSEC (kJ/kg)	R^2_p	RMSEP(kJ /kg)	RPD	bias (kJ/kg)
Full-PLSR	First derivative(g = 5, s= 5)	15	0.9694	132.4790	0.9578	186.0301	4.8759	9.2028
GA-PLSR	First derivative (SW = 176)	12	0.9545	161.6585	0.9498	203.0148	4.4629	-2.5835
SPA-PLSR	First derivative (SW = 1139)	15	0.9594	132.4765	0.9478	186.0129	4.7763	9.1919
MP-PLSR:3 range	Combination set: 0,5,4	7	0.8977	242.2838	0.8702	326.4044	2.8067	-48.4726
MP-PLSR: 5 range	Combination set: 2,5,2,0,5	14	0.9223	211.1559	0.9212	254.3010	3.6269	-47.6977

The model's performance on the validation set, assessed following guidelines by Williams, *et al.* [22] for R^2_p and Zornoza, *et al.* [23] for RPD, indicates that the selected model, Full-PLSR with preprocessing from the first derivate, can be applied in various applications, including quality assurance, and demonstrates excellent prediction capabilities.

Similarly, 74 biomass samples collected from the second batch were scanned in an FT-NIR spectrometer under the same laboratory conditions, and their HHV was evaluated. These samples were used as an unknown sample set. The performance of the unknown sample set was evaluated in terms of R^2_{UN} , RMSEUN, RPD_{UN}, and BiasUN, resulting in values of 0.8089, 360.8845 kJ/kg, 2.44, and -131.4356 kJ/kg, respectively. The model's performance is comparatively lower for the unknown sample set compared to the validation set. This discrepancy could stem from differences in biomass characteristics, environmental conditions during sample collection, and variations in sample preparation. Also, seasonal variations in plant growth may influence the chemical composition of the biomass, thereby affecting its HHV. Based on the performance of the unknown sample set, following Williams, *et al.* [22] and Zornoza, *et al.* [23] the model can make approximate quantitative predictions for screening and other appropriate calibrations.

Figure 4 displays the measured and predicted values of calibration, validation, and unknown samples for the Higher Heating Value (HHV) of biomass obtained using Full-PLSR. In the figure, the trend lines for the calibration and validation sets overlap, indicating similar predictability. However, the trend line for the unknown sample set shows an offset compared to the trend lines for the calibration set and the validation set, as well as the 45-degree target line. This offset raises concerns about the model's performance with the unknown sample set. Therefore, by existing NIRS protocol,

the unknown set and validation set have to be input into updating model to be used in future for higher robustness of the model specifically.

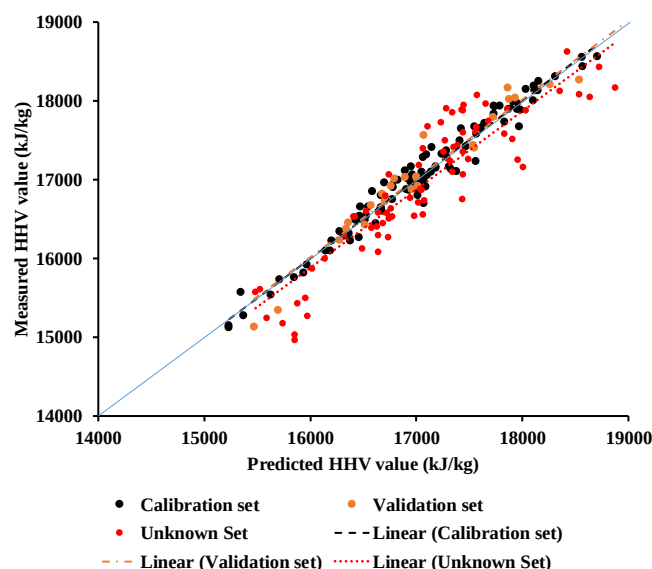


Figure 4. Measured versus predicted values in the calibration, validation and unknown sample sets for the HHV of the biomass.

Figure 5 shows the regression coefficient plot for HHV (kJ/kg) of the biomass, which is obtained from the first derivative preprocessing Full-PLSR model. The significant peaks were observed at wavenumbers 3733, 4525, 4762, 5155 and 11655 cm^{-1} which might have significant influence in the performance of the model. Interpreted by Workman and Weyer [24]: the positive peak at 3733 cm^{-1} corresponds to the functional group of C-H aromatic with material types as C-H aryl. Similarly, the negative peak at 4525 cm^{-1} corresponds to the spectral-structure of the second overtone of fundamental stretching band of N-H for NH_3 in water, and functional group of N-H ammonia in water. The peaks at 4762 cm^{-1} are associated with spectra structure of combination of O-H bending and C-O stretching with material type as polysaccharides. The peaks at 5122 cm^{-1} are associated with combination of O-H stretching and HOH bending with material type as a water. The peak at 11655 cm^{-1} corresponds to the third overtone of fundamental stretching band of C-H with material type as hydrocarbons, aromatic. These findings provide valuable insights into the spectral features contributing to the prediction of HHV in biomass.

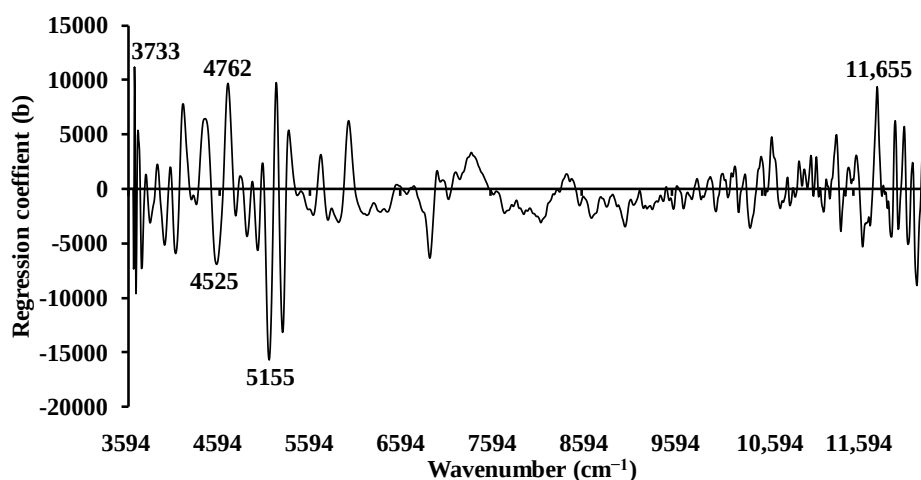


Figure 5. The regression coefficient for the HHV (kJ/kg) of biomass using the Full-PLSR.

Table 3 shows the performance of updating model created by using every sample spectra in full wavenumber range of calibration set, validation set and unknown set pretreated by first derivative ($g = 5, s = 5$) and corresponding HHV and subjected to PLSR. The result shows that when the LVs was 15 as same as the selected model of the best performance, the R^2_{cv} was 0.95 and RMSECV and SECV was very closed to each other. Therefore, the average error or bias was very closed to zero. R^2_{cv} of 0.95 and RPD_{cv} was 4.6 indicating that the model was excellent for any application including HHV of biomass quantification. This is proved for the updating model can be the base model for practical implementing in biomass industries.

Table 3. Results of the updating PLSR-based model for the HHV (kJ/kg) of biomass ready to be implemented in biomass industries, with best performing model highlighted in bold.

LVs	R^2_{cv}	RMSECV	SECV	RPD _{cv}	LVs	R^2_{cv}	RMSECV	SECV	RPD _{cv}
1	0.1571	797.41	799.71	1.1	8	0.8284	359.75	360.79	2.4
2	0.2441	755.14	757.32	1.2	9	0.8556	330.06	331.01	2.6
3	0.3662	691.44	693.44	1.3	10	0.9021	271.70	272.49	3.2
4	0.5198	601.84	603.57	1.4	11	0.9207	244.65	245.36	3.6
5	0.6233	533.06	534.60	1.6	12	0.9309	228.31	228.97	3.8
6	0.7348	447.29	448.58	1.9	13	0.9417	209.76	210.37	4.1
7	0.7972	391.11	392.23	2.2	14	0.9470	199.98	200.56	4.3
					15	0.9516	191.03	191.58	4.6

4. Discussion

Two general classes of spectrometers including wavelength-dispersive and Fourier transform (FT), where the former, the wavelength selector only passes selected, narrow wavelength windows that can reach detector at a time [25]. By FT principle, an interferometer (mostly Michelson interferometer is employed) enables a simultaneous incidence of all wavelengths on the detector and spectrum is obtained through Fourier transform [25]. The FTIR spectrometers have the well-known advantages of the approach: "Fellgett advantage": Multiplex operation, i.e. all wavelengths are captured simultaneously, resulting in short scan times; "Jacquinot advantage": High optical throughput as no slits limit the aperture, resulting in a good signal-to-noise ratio (SNR); "Connes advantage": Linearity of the wavelength scale can easily be calibrated, e.g. using a reference laser [26].

The NIR spectra obtained by FT-NIR spectrometer with full wavenumber range of 12500-4000 cm^{-1} combined with PLSR for HHV of biomass prediction model development is shown in some researches, for example, rice husk [27], ground sorghum biomass [28], wood sawdust from *Eucalyptus benthamii* Maiden & Cabbage, *Eucalyptus dun nii* Maiden and *Eucalyptus saligna* Sm [29], ground bamboo [30], bamboo wood chip [31], biomass made up of needles, twigs, branches, bark and wood of *Pinus taeda* (loblolly pine) [32], ground biomass of fast growing trees and agricultural residue [33], and ground cassava rhizome [34] in which the R^2_P were 0.79, 0.96, 0.98, 0.92, 0.84, 0.34, 0.96, and 0.90, respectively. As expected, the homogeneous of ground biomass models provide better R^2_P due to less noise in scattering. Although ground sample described a better performance, the operation in the power plant was found to be inconvenient due to sample preparation costs and labour required for the necessary preparation of ground samples [31].

However, in case of our result of model performance for wood chip biomass of fast growing trees and agricultural residue show better R^2_P of 0.96 and R^2_{UN} of 0.81 which indicating outperform of the model to bamboo wood chip model (R^2_P of 0.84) which contained only one specie. It was noticed for model for biomass made up of needles, twigs, branches, bark and wood of *Pinus taeda* (loblolly pine) [32] which provided very low R^2_P indicating the cause and effect must be researched which, for example, might be due to heterogeneous sample while scanning, though same specie when other models mentioned before were scanned on more homogeneous with smaller particle size of same specie. The FTIR spectra (4000-650 cm^{-1}) of whole tree, wood and bark, slash (i.e., limbs and foliage), and clean wood chips in ground form of several loblolly pine plantations combined with PLSR

provided R^2_P by cross validation of 0.64 [35], though not well but very better than, by NIR spectroscopy (R^2_P of 0.34) [30] obviously. By 400-750 nm visible spectra of *Eucalyptus* sawdust obtained by spectrophotometer, PLSR model provided R^2_P of 0.985 [29] and by 10000-4000 cm^{-1} FT-NIR spectra of the same sample, PLSR model provided better R^2_P of 0.9779 [29]. There was very few reports on combined different species of biomass in one model like we did. Additionally without any other reports, our updating PLSR model for HHV of wood chip of fast growing trees and agricultural residue was proved for capability to implement for biomass industrial use.

5. Conclusions

The rapid non-destructive modeling created by FT-NIR sensor confirmed its phenomenon in biomass energy prediction in this work. It is proved that wood chip Nepali biomass prediction model developed for evaluation of HHV of the biomass is ready to be installed in the biomass industry for updating in the industrial environment by developing the robust model combining the calibration, validation, and unknown sets. This finding is not only useful to biomass industry but for other various stakeholders including biomass traders, engineers and researchers involved in the design and development of energy system, for example, in combustion via combined heat and power systems, and renewable energy policymakers.

Author Contributions: B.S.: conceptualization, methodology, software, formal analysis, investigation, resources, data curation, visualization, writing the original draft, writing—review and editing. T.P.: software, formal analysis. Z.S.: investigation. J.P.: conceptualization, methodology, software, formal analysis, data curation, writing—review and editing, supervision. P.S.: conceptualization, methodology, data curation, writing the original draft, writing—review and editing, validation, supervision, project administration, funding acquisition. B.P.S.: conceptualization, methodology, writing—review and editing, project administration, and supervision. P.P.: conceptualization, methodology, writing—review and editing, and supervision. H.A.: writing the original draft, writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data will be made available upon request from the corresponding author.

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