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A Correlation for Estimating Elemental Composition of Lignocellulosic Biomass from Its Volatile and Fixed Solids Content

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Abstract: The elemental composition of a lignocellulosic biomass is a significant property, which describes its energy value and helps in decision for its efficient use. Its experimental determination is an expensive process, while the determination of volatile solids (VS) and fixed solids (FS) needs only standard laboratory muffle furnace and can be operated with a very less skill. The aim of present work is to determine correlations for estimating mass fractions of C, H, O, N and S for lignocellulosic biomass from their VS and FS content. The correlations were developed through nonlinear multiple regression analysis of experimental data and their average absolute errors and the average bias errors were also calculated. Acquiring elemental composition from developed correlations is simple, economical, less time consuming technique. The other benefits of these correlations include a convenient modeling tool in combustion, bio-gasification, thermal gasification and pyrolysis processes.

Keywords: elemental composition, ultimate analysis, lignocellulosic biomass, volatile solids, fixed solids.

1. INTRODUCTION

All living or recently living material is known as biomass, which is organic in nature. Biomass stores the solar energy in the form of chemical bonds. At time, when chemical bonds fragmented by any chemical or biological process, the stored energy in the biomass get released (McKendry, 2002). Biomass accounts about 14% of world's energy consumption, 25% of which is used by developed countries, while the rest of 75% is used by developing country to produce heat (Parikka, 2004). Biomass is generally an animal or plant material, used to provide food, fodder, fertilizer, heat and electricity (Sahito *et al.,* 2012). Lignocellulosic biomass is that type of biomass having resistance to degrade biologically and consist of cellulose, hemicellulose and lignin. Lignocellulosic biomass types include woody biomass (forest residues, wood chips, and saw dust) and non-woody biomass (wheat straw, sugarcane bagasse, canola straw, etc.).

The elemental composition of lignocellulosic biomass is usually ascertained by elemental analysis, but can also be estimated through empirical equations, which are based on experimental data. The elemental analysis of lignocellulosic biomass comes within the domain of analytical chemistry, and mostly referred as the determination of the mass fractions of carbon, hydrogen, nitrogen, and sulfur present in the sample. The elemental analysis is accomplished by burning the sample in an excess of oxygen gas, whereas the products of combustion are trapped and measured by Gravimetric method. The mass fraction of oxygen is usually determined as the difference of total mass of sample to the mass fractions of carbon, hydrogen, nitrogen, and sulfur (Telmo *et al.,* 2010).

The estimation of elemental composition of lignocellulosic biomass can also be made by using empirical equations based on the proximate analysis (Parikh *et al.,* 2007 and Shen *et al.,* 2010). The proximate analysis is used to demonstrate the combustible to incombustible constituents of a sample and involves the determination of the mass fractions of moisture content (MC), volatile matter (VM), ash content (AC) and fixed carbon (FC) (Tchobanoglous *et al.,* 2000). The proximate analysis is carried out by using muffle furnace (Erol *et al.,* 2010). In proximate analysis the MC of lignocellulosic biomass sample is calculated as the mass difference before and after heating it at boiling temperature of water. The *VM* is determined by further heating the sample at 950 °C in inert atmosphere. The AC is determined by burning the sample at 750 °C to 815 °C, whereas the *FC* is estimated by subtracting *AC* from nonvolatile fraction of sample (Parikh *et al.,* 2005).

Besides proximate analysis, the volatile solids (VS) are also determined by gravimetric method. The VS content is the fractions of biomass, which can be volatized at 550°C and is frequently used as the measure of organic fraction of biomass. The fixed solids (FS) is the mass fraction of biomass residue remained after furnace dried sample is ignited and oxidized at 550 °C (Tchobanoglous *et al.,* 2003). Regardless of the speedily increasing literature in the field of, no any effort has been made to find the correlations to estimate elemental composition from *VS* and *TS* of biomass. Determination of *VS* not only gives the mass fraction of organic material present in the biomass but is less time and energy consuming process than to the *VM*, because of lower temperature involvement in case of *VS*. Keeping

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in view above dual advantage of *VS* determination, the present study is carried out to establish correlations for estimating percentages of elemental carbon (C), hydrogen (H), oxygen (O), nitrogen (N) and sulfur (S) for lignocellulosic biomass from their *VS* and *FS* contents.

2. MATERIAL AND METHODS

2.1 Characteristics of lignocellulosic biomass

The representative samples of eleven lignocellulosic biomass i.e. sugarcane bagasse, banana plant trash, canola straw, corn cob, cotton gin waste, sugarcane trash, cotton stalks, rice husk, rice straw, saw dust and wheat straw were collected from the agricultural field and after drying and crushing, they were analysed for *TS, VS, FS, AC, C, H, O, N* and *S*. The *TS* content was ascertained by employing ASTM standard method E1756-08 (ASTM Standard E1756, 2008). The mass fraction of VS content was determined by ignition of oven dried samples in inert atmosphere at 550 °C. The *FS* is the residue left after igniting the sample at 550 °C and was calculated by deducting mass fraction of *VS* from mass fraction of *TS*. The *AC* was ascertained by employing standard method ASTM E1755-01 (ASTM Standard E1755-01, 2007). The ultimate analysis was carried out by using standard method ASTM D3176 (ASTM Standard D3176-09, 2009). The mass fractions of elemental *C, H, N* and *S* were ascertained by using elemental analyser, whereas the mass fraction of *O* was calculated as the difference of mass fractions of elemental *C, H, N, S* and *AC* from total mass of sample (Telmo *et al.*, 2010). The characteristics of lignocellulosic biomass are given in Table 1.

2.2 Development of correlations

The development of new correlations was based on the oven dried mass fractions of *VS* and *FS*, which estimates the elemental *C, H, O, N* and *S*, present in the dried sample of lignocellulosic biomass. The *VS* represents the quantity of the organic matter present in the sample, while the *FS* represents the quantity of the inorganic combustible solids and *AC*. Both the *VS* and *FS* contain the mass fractions of elemental *C, H, O, N* and *S*, thus it was assumed that the new correlations are the functions of the *VS* and *FS*. Afterwards, following six criteria were assumed that the elemental *C, H, O, N* and *S* are the:

1. Linear functions of *VS* and *FS*, mathematically;
$$y = a \times VS + b \times FS \quad (1)$$
2. Linear functions of *VS* and nonlinear functions of *FS*, mathematically;
$$y = a \times VS + b \times FS^{c} \quad (2)$$
3. Nonlinear functions of *VS* and linear functions of *FS*, mathematically;
$$y = a \times VS^{b} + c \times FS \quad (3)$$
4. Nonlinear functions of *VS* and *FS*, mathematically;
$$y = a \times VS^{b} + c \times FS^{d} \quad (4)$$
5. Multiple polynomial functions of two variables i.e. *VS* and *FS*, mathematically;
$$y = a_1 \times VS^{n} + b_1 \times VS^{(n-1)} + \cdots + z_1 + a_2 \times VS^{n} + b_2 \times VS^{(n-1)} + \cdots + z_2 \quad (5)$$
6. Multiple inverse polynomial functions of two variables i.e. *VS* and *FS*, mathematically;
$$y = a_1 \div VS^{n} + b_1 \div VS^{(n-1)} + \cdots + z_1 + a_2 \div VS^{n} + b_2 \div VS^{(n-1)} + \cdots + z_2 \quad (6)$$

Table 1: Characteristics of lignocellulosic biomass

Name of Biomass	wt. % sample		wt. % on dry basis		wt. % on dry basis					
	MC	*TS*	*% VS*	*% FS*	*% AC*	*% C*	*% H*	*% O*	*% N*	*% S*
Wheat straw	4.3	95.7	64.1	35.9	14.6	40.8	5.3	38.1	0.8	0.2
Sugarcane trash	2.4	97.6	66.4	33.6	13.6	39.9	3.7	42.5	0.2	0.1
Sugarcane bagasse	3.1	96.9	75.4	24.6	4.7	44.7	5.5	44.9	0.2	0.0
Saw dust	7.7	92.3	69.0	31.0	6.8	44.4	5.1	43.2	0.4	0.1
Rice straw	3.3	96.7	62.3	37.7	18.8	36.4	5.0	38.7	1.0	0.2
Rice husk	4.8	95.2	61.4	38.6	20.6	36.9	5.6	35.1	1.7	0.2
Maize cob	4.6	95.4	72.0	28.0	7.0	44.7	5.7	41.5	1.0	0.1
Cotton stalks	4.8	95.2	63.5	36.5	10.0	42.8	5.6	41.1	0.1	0.4
Cotton gin waste	4.6	95.4	60.8	39.2	20.8	39.9	5.1	32.0	1.7	0.6
Canola straw	9.1	90.9	68.6	31.4	9.5	39.7	5.3	44.4	0.2	0.9
Banana plant trash	6.0	94.0	62.7	37.3	16.7	38.3	5.4	39.2	0.4	0.1

Where *y* is the mass fraction of elemental *C, H, O, N* or *S* and *a, b, c, d, a_1, a_2, b_1, b_2, …* are the coefficients of the proposed correlations and *n* is order of polynomial, which ranges from 1 to 10. The

coefficients of each correlation, based on the assumed criteria for elemental *C, H, O, N* and *S* were estimated by carrying out non–linear multiple regression using least square method for their best fit values.

2.3. Discrimination in developed correlations

The The accuracy evaluation of the proposed correlations were accomplished by assessing the coefficient of multiple determinations *(R^2)* and root mean square error *(RMSE)* between the experimental and estimated values of elemental composition. The correlation makes a better estimation with a higher value of R^2 and lowest value of *RMSE*. The R^2 was calculated Eq. 7 (Friedl *et al.,* 2005), whereas the *RMSE* was calculated through Eq. 8 (El-Mashad, 2013).

$$R^2 = 1 - \frac{\sum_{j=1}^{m}(X_{Exp,j} - X_{Est,j})^2}{\sum_{j=1}^{m}(X_{Exp,j} - \overline{X}_{Est,j})^2} \quad (7)$$

$$RMSE = \sqrt{\frac{\sum_{j=1}^{m}(X_{Exp,j} - X_{Est,j})^2}{m}} \quad (8)$$

Where, X_{Exp} is the experimental, and X_{Est} is the estimated values of elemental composition; whereas *m* is the number of data points. Additionally, developed correlations were also evaluated through their average absolute error (AAE) and average bias error (ABE) by employing Eq. 9 and 10, respectively.

$$AAE = \frac{1}{n}\sum_{j=1}^{m}\left|\frac{X_{Exp,j} - X_{Est,j}}{X_{Est,j}}\right| \times 100 \quad (9)$$

$$ABE = \frac{1}{n}\sum_{j=1}^{m}\left[\frac{X_{Exp,j} - X_{Est,j}}{X_{Est,j}}\right] \times 100 \quad (10)$$

The lowest value of *AAE* reflects the highest accuracy of the correlation, whereas the negative and positive values of the *ABE* indicate the underestimation or overestimation of the correlation (Sheng *et al.,* 2005 and Yin, 2011).

3. RESULTS AND DISCUSSION

3.1 Correlation for Carbon

The statistical analysis of the developed correlations for carbon is given in Table 2. Regarding the R^2, *RSME, AAE* and *ABE* values, the Eqs. 1 to 4 have about same accuracy and not exactly follow the assumption made for their development that the carbon is the linear/ nonlinear function of the VS and FS. However, highest accuracy was obtained for Eq. 6 followed by Eq. 5, which shows that the carbon content present in the lignocellulosic biomass follow the two variable multiple inverse polynomial well. The accuracy of Eqs. 1 to 4 was on the lower side because percentage of carbon in the lignocellulose biomass is not linear nor nonlinear with the percentages of *VS* and *FS* as in case of volatile matter and fixed carbon content (Cordero *et al.,* 2001). Additionally, discussing to the negative values of the *ABE*, all the equations underestimate the values of carbon except Eq. 5.

Table 2: Statistical analysis of developed correlations for carbon

Element	Eq. No.	R^2	*RSME*	*AAE*	*ABE*
Carbon	Eq.1	0.594	1.833	4.0924	-0.0009
	Eq.2	0.595	1.832	4.0880	-0.0017
	Eq.3	0.595	1.831	4.0841	-0.0008
	Eq.4	0.595	1.832	4.0874	-0.0014
	Eq.5	0.716	1.534	3.1518	0.3898
	Eq.6	0.924	0.792	1.3141	-0.0004

3.2 Correlation for Hydrogen

The statistical analysis of the developed correlations for hydrogen is presented in Table 3. In regard to the R^2 values, the Eqs. 1 to 4 violates the assumption that the hydrogen is the linear/ nonlinear function of the *VS* and *FS*. The accuracy of Eq. 5 for hydrogen content in lignocellulosic biomass is also very low. On the contrary, the mass fraction of hydrogen present in the lignocellulosic biomass can be estimated well by using Eq. 6. Moreover, mentioning to the values of the ABE, Eq. 2, Eq. 4 and Eq. 5 underestimate the values of hydrogen, whereas other equations overestimates.

Table 3: Statistical analysis of developed correlations for hydrogen

Element	Eq. No.	R^2	*RSME*	*AAE*	*ABE*
Hydrogen	Eq.1	0.011	0.522	6.5180	0.0004
	Eq.2	0.043	0.513	6.5388	-0.0129
	Eq.3	0.013	0.521	6.5155	0.0085
	Eq.4	0.047	0.242	6.5645	-0.0038
	Eq.5	0.619	0.324	4.6538	-0.8324
	Eq.6	0.969	0.092	1.2720	0.0058

3.3 Correlation for Oxygen

The statistical analysis of the developed correlations for oxygen is presented in Table 4. Regarding the R^2, *RSME, AAE* and *ABE* values, the Eqs. 1 to 4 follows the assumption that the oxygen is the linear/ nonlinear function of the *VS* and *FS*. Especially, Eq. that has the R^2 value of 0.772 depict that the oxygen content in the lignocellulosic biomass is the nonlinear function of it *VS* and *FS* contents. However, highest accuracy was obtained for Eq. 6 followed by Eq. 5. Besides, referring to the negative values of the *ABE*, all the equations underestimate the values of oxygen except Eq. 6.

Table 4: Statistical analysis of developed correlations for oxygen

Element	Eq. No.	R^2	*RSME*	*AAE*	*ABE*
Oxygen	Eq.1	0.638	2.275	5.1194	-0.0190
	Eq.2	0.715	2.018	4.4407	-0.0704
	Eq.3	0.642	2.263	5.0951	-0.0195
	Eq.4	0.772	1.805	4.0111	-0.0135
	Eq.5	0.952	0.829	1.4707	-0.0006
	Eq.6	0.978	0.559	0.9108	0.0001

3.4 Correlation for Nitrogen

The statistical analysis of the developed correlations for nitrogen is expressed in Table 5. Considering the values of R^2, *RSME, AAE* and *ABE*, the Eqs. 1 to 4 violates the assumption that the nitrogen is the linear/ nonlinear function of the VS and FS. On the contrary, the mass fraction of nitrogen present in the lignocellulosic biomass can be estimated well by using Eq. 6 followed by Eq. 5. Nevertheless, Eqs. 5 and 6 produces higher values of *AAE*, even than the nitrogen content present in the lignocellulosic biomass follow the two variables multiple inverse polynomial well as they have highest value of R^2 and lowest value of *ABE*. Furthermore, referring to the positive values of the *ABE*, all the equations overestimate the values of nitrogen except Eq. 6.

Table 5: Statistical analysis of developed correlations for nitrogen

Element	Eq. No.	R^2	*RSME*	*AAE*	*ABE*
Nitrogen	Eq.1	0.249	0.487	66.1652	11.2352
	Eq.2	0.288	0.475	64.9956	12.1476
	Eq.3	0.250	0.487	66.2912	11.4172
	Eq.4	0.300	0.471	65.7845	13.8568
	Eq.5	0.831	0.231	40.1008	13.5313
	Eq.6	0.940	0.138	19.5089	-1.6855

3.5 Correlation for Sulfur

The statistical analysis of the developed correlations for sulfur is given in Table 6. Looking to the values of R^2, *RSME, AAE* and *ABE*, the Eq. 4 does not converges, whereas the Eqs. 1 to 3 significantly violates the assumption that the sulfur is the linear/ nonlinear function of the VS and FS. The accuracy of Eq. 5 for sulfur content in lignocellulosic biomass is also very low as it has very high values of *AAE* and *ABE* of about 142 and -71, receptively. On the contrary, the mass fraction of sulfur present in the lignocellulosic biomass can be estimated well by using Eq. 6, thus establishes that sulfur content present in the lignocellulosic biomass follow the two variable multiple inverse polynomial well. Moreover, referring to the negative values of the *ABE*, all the six equations underestimate the values of sulfur.

Table 6: Statistical analysis of developed correlations for sulfur

Element	Eq. No.	R^2	*RSME*	*AAE*	*ABE*
Sulfur	Eq.1	0.066	0.248	76.6567	-2.4608
	Eq.2	0.068	0.248	76.4909	-2.7430
	Eq.3	0.086	0.246	74.8548	-5.5017
	Eq.4	Not converges			
	Eq.5	0.616	0.159	142.535	-71.454
	Eq.6	0.965	0.048	24.2239	-6.9400

3.6 Best fit correlations for elemental composition of lignocellulosic biomass based on VS and FS content

The above discussion reveals that the elemental composition of lignocellulosic biomass can be estimated through the two variables multiple inverse polynomial correlations. From the results of the non–linear multiple regression analysis the elemental *C*, *O* and *N* follow the Eq. 11 and elemental *H* and *S* follows the Eq. 12. The coefficients of Eqs. 11 and 12 are given in Table 7.

$$y = a + b \div VS + c \div VS^2 + d \div VS^3 + e \div FS + f \div FS^2 + g \div FS^3 + h \div FS^4 + i \div FS^5 \quad (11)$$

$$y = a + b \div VS + c \div VS^2 + d \div VS^3 + e \div VS^4 + f \div FS + g \div FS^2 + h \div FS^3 + i \div FS^4 \quad (12)$$

Table 7: Values of coefficients for Eq. 11 and 12

Coefficients	Carbon	Hydrogen	Oxygen	Nitrogen	Sulfur
a	-769836432	-913648400	-354084180	185638310	-541784822
b	53336590888	132518463892	26890368860	-13748604308	77123893884
c	-2052568816336	-8941141094152	-1075775995120	544691186460	-5143869824388
d	29007115595517	285172523422012	15707602791583	-7891299768369	162362384209635
e	28616168177	-3547834191931570	11388477283	-6239351983	-2000323465248460
f	-1045738795334	9724235745	-389106923267	218037175352	6060480432
g	21630980103168	-217430907734	7546374637609	-4325892811179	-137579800770
h	-240716463389841	2545003967511	-78833548294295	46260107022829	1632476853345
i	1125116010237190	-12313989659713	346094717621267	-208070145465103	-7998782442648

These correlations are valid for lignocellulosic biomass, having *VS* content between 60.8–75.4%, *FS* content between 24.6–39.2%, ash content between 4.7–20.8%, carbon content between 36.4–44.7%, hydrogen content between 3.7–5.7%, oxygen content between 32.0–44.9%, nitrogen content between 0.1–1.7% and sulfur content between 0–0.9 percent in wt. % on dry basis. The developed correlations have 1.3%, 1.3%, 0.9%, 19.5%, 24.2% of average absolute errors and have the -0.0004%, -0.0004%, 0.0058%, 0.0001%, -1.69%, -6.94% of average bias errors in comparison to the experimental values of elemental *C*, *H*, *O*, *N* and *S* respectively. In comparison to the correlations available in literature based on the proximate analysis (Parikh *et al.*, 2007 and Shen *et al.*, 2010), the correlation developed on the basis of *VS* and *FS* have lower values of *AAE* and *ABE* and thus give more accuracy.

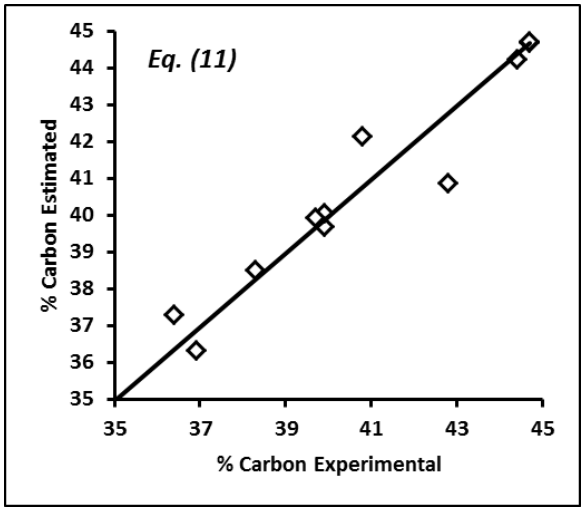


Fig. 1: Comparison between experimental and estimated values of carbon

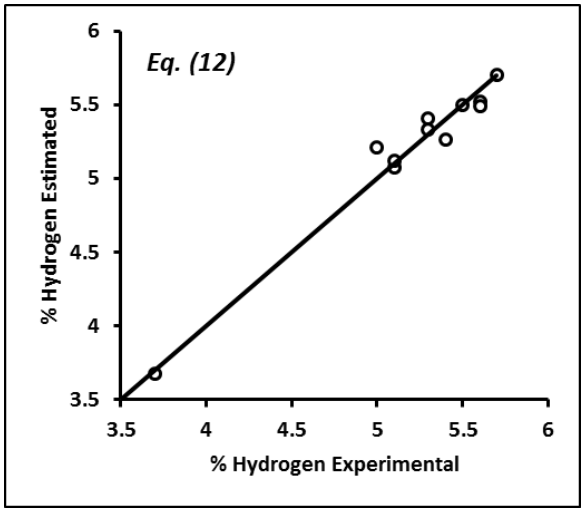


Fig. 2: Comparison between experimental and estimated values of hydrogen

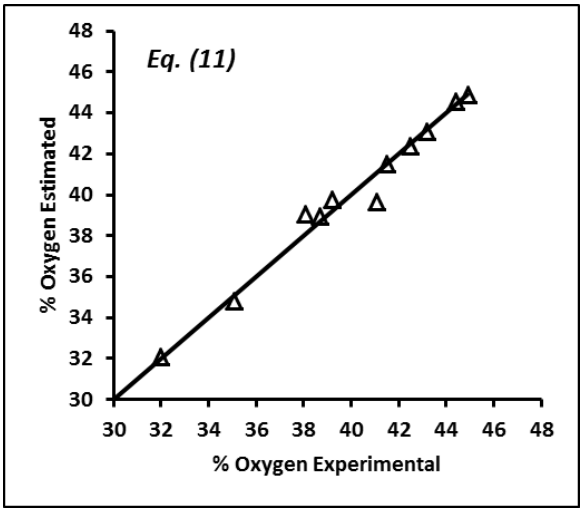


Fig. 3: Comparison between experimental and estimated values of oxygen

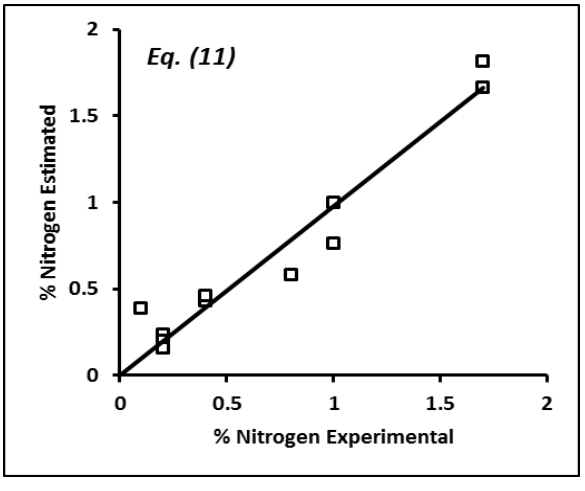


Fig. 4: Comparison between experimental and estimated values of nitrogen

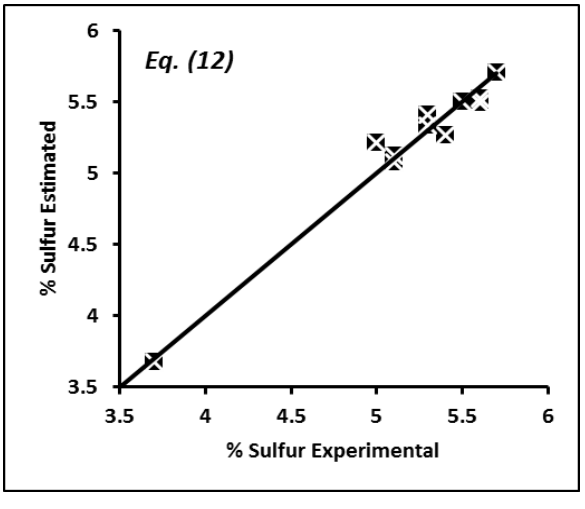


Fig. 5: Comparison between experimental and estimated values of sulphur

Moreover, the comparison of experiential elemental composition of lignocellulosic biomass with the estimated through the developed Eqs. 11 and 12 is shown in Fig. 1-5. These graphs are drawn by setting intercept to zero and depicts that the experimental values of the elemental composition of lignocellulosic biomass are exceptionally fit to the estimated values.

4. CONCLUSIONS

The experimental determination of elemental composition of biomass is an expensive process. On the contrary, the determination of volatile solids and fixed solids is a cheap process. The present work yields the correlations for estimating dried mass fractions of carbon, hydrogen, oxygen, nitrogen and sulfur for lignocellulosic biomass from their volatile and fixed solids content. The correlations were developed through nonlinear multiple regression analysis of experimental data. Moreover, the correlations were statistically discriminated through the coefficient of multiple determinations, root mean square errors, average absolute errors and the average bias errors. The present study concludes that the elemental composition of the lignocellulosic biomass is the linear function of its volatile and fixed solids content, and follows the two variables multiple inverse polynomial well. The correlations are valid for lignocellulosic biomass, having volatile solids content between 60.8–75.4%, fixed solids content between 24.6–39.2%, ash content between 4.7–20.8%, carbon content between 36.4–44.7%, hydrogen content between 3.7–5.7%, oxygen content between 32.0–44.9%, nitrogen content between 0.1–1.7% and sulfur content between 0–0.9 percent in wt. % on dry basis. The developed correlations have 1.3%, 1.3%, 0.9%, 19.5%, 24.2% of average absolute errors and have the -0.0004%, -0.0004%, 0.0058%, 0.0001%, -1.69%, -6.94% of average bias errors in comparison to the experimental values of elemental *C, H, O, N* and *S*, respectively. In comparison to the correlations available in literature based on the proximate analysis, these correlations have lower error values and thus give more accuracy. Moreover, acquiring elemental composition of lignocellulosic biomass from developed correlations is not only simple, economical, less time consuming technique, but is a convenient modeling tool in combustion, bio-gasification, thermal gasification and pyrolysis processes.

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Nomenclature

a, b, c, d, e, f, g, h, i, a_1, a_2, b_1, b_2, …	Coefficients of the proposed correlations
AAE	Average absolute error (%)
ABE	Average bias error
m	Number of data points
n	Order of polynomial
R^2	Coefficient of multiple determinations
RSME	Root mean square error (%)
X_{Est}	Estimated elemental composition (%)
X_{Exp}	Experimental elemental composition (%)
y	Mass fraction of elemental *C, H, O, N* or *S*

Abbreviations

°C	Degree Celsius
AC	Ash content
ASTM	American Society for Testing and Materials
C	Elemental carbon
FC	Fixed carbon
H	Elemental hydrogen
MC	Moisture content
N	Elemental nitrogen
O	Elemental oxygen
S	Elemental sulfur
TS	Total solids
TS	Total solids
VM	Volatile matter
VS	Volatile solids

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