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Influence of microwave-assisted chemical treatment on delignification of rice straw biomass

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Abstract

Lignocellulosic biomass (like rice straw) provides an alternative for depleting non-renewable energy sources through its value-added utilization (like production of biofuels and nanocellulose) owing to its abundance, renewability, polymer presence and environmental friendliness. Prior to its utilization, any lignocellulosic biomass is subjected to a time-consuming delignification process for lignin free biomass recovery. The present study aims to reduce the time of delignification of rice straw along with enhancing the delignification percentage of biomass by use of microwave assisted sodium chlorite method. The experiments were done at two microwave power levels (640, 800 W), three bleaching solution concentrations (0.4, 1.7, 3.0 %) and three microwave treatment times (4, 8, 12 min). The delignification percentage of the rice straw for the whole experimentation varied from 24.7 to 90.12%. The results revealed that the time of delignification was greatly reduced (12 min) with a very high delignification (90.12%) percentage. The morphology of the delignified samples also revealed the deconstruction of the lignin structure. The improved method can thus be applied for the delignification of other biomasses as well for quick and effective delignification.

Key words: ***Delignification Percentage, Bleaching, Lignocellulosic biomass, Microwave assisted chemical treatment, Rice straw, Sodium Chlorite***

Introduction

In the contemporary world of environmental concern due to fossil fuel depletion and increased carbon footprint (Sain *et al*., 2020), more emphasis has been laid on conventional and value-added non-conventional utilization of lignocellulosic biomass (Saratale, 2012). Lignocellulosic biomass refers to the plant dry matter which plays an important role in the carbon cycling process. It embodies a wide spectrum of materials ranging from agricultural wastes, forest residues, industrial by-products, to energy crops, primary crop residues, etc. with a global annual production of 181.5 billion tons and energy value between 5 and 50 EJ/year (Kang *et al*., 2014; Maurya *et al*., 2015; Paul and Dutta, 2018). Conventionally, lignocellulosic biomass has been used for the production of biofuels, bioethanol, grafted polymers, platform chemicals apart from its unscientific burning which causes secondary environmental pollution (Bozell *et al*., 2011; Kang *et al*., 2014; Mukherjee *et al*., 2018; Zheng *et al*., 2014; Sain, 2020). With the advent of nanoscience and nanotechnology, a revolutionary concept, the lignocellulosic biomass has been utilized non-conventionally by its miniaturization to nano-scale (Lee *et al*., 2014; Yu *et al*., 2021). Before both conventional and non-conventional utilization of lignocellulosic biomass, delignification is the most important step for obtaining lignin-free holocellulose (cellulose and hemicellulose) (Jin *et al*., 2019). Several methods have been reported in the literature for the delignification of lignocellulosic biomass (Kucharska *et al*., 2018). The still considered and the most effective method is the acidified sodium chlorite delignification (Kumar *et al*., 2013). However, the procedure is time-consuming and the lignin is not removed completely in most of cases due to the recalcitrant nature of the lignocellulose. To speed up the process and enhance lignin removal, microwave-assisted delignification has been used (Kohli *et al*., 2020; Liu *et al*., 2017). Microwaves possess innate ability in presence of moisture (dipoles or ions) to open up the structure of the cell walls and make the components available for subsequent reaction (Nour *et al*.,

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2021). Rice stands to be the largest produced cereal crop globally, with a production of 782 million tonnes in 2018 and increasing by almost 1.8 % yearly. (FAOSTAT, 2020). In rice production, the volumetric percentage of rice straw is about 45%, and is quantitatively the highest crop residue (Lu and Hsieh, 2012). Each kg of milled rice produced results in roughly 0.7–1.4 kg of rice straw depending on varieties, cutting height of the stubbles, and moisture content during harvest (IRRI, 2020). Rice straw residue has been utilized for the production of biofuels, grafted polymer fermentable sugars as well as for production of novel nanocellulose preceded by an essential delignification step, which can be enhanced by microwaves to improve the purity of final product (Lu and Hsieh, 2012; Mukherjee *et al.*, 2018; Oun and Rhim, 2016, 2018). The present study was aimed to improve the speed and effectiveness of delignification of rice straw biomass by microwave-assisted chemical treatment.

Materials and Methods

The experiments were carried out at the Department of Post-Harvest Technology, GBPUAT, Pantnagar, Uttarakhand, India. Rice straw was collected from the Crop Research Centre of the university. Standard chemicals including ethanol, toluene, sodium chlorite, and glacial acetic acid, nitric acid were used in the experiment.

Experimental Procedure

The initial composition of the rice straw viz. moisture content, oven-dry solids, extractives, and lignin were determined using standard procedures reported in the literature (Sluiter *et al.*, 2008a; Sluiter *et al.*, 2012; Sluiter *et al.*, 2008b). The biomass recovered was calculated based on the weight difference method (initial solid weight minus extractives and lignin). The experimentation was done using dewaxed rice straw powder (RSP) of a particular size. The dewaxing was done for extractive removal as reported by (Lu and Hsieh, 2012).The rice straw was first thoroughly washed with tap water to remove dust particles before being washed with distilled water. This was followed by drying at 105 °C for 24 hrs. The dried rice straw was then ground using a hammer mill and sieved to get a powder size of 300 µm. 30 g rice straw powder (RSP) was dewaxed (by refluxing method) with a 450 ml mixture of ethanol/toluene (1:2) for 20 h. This was followed by vacuum filtration of the sample and subsequent drying in the oven at 50 ℃ for 5 h to remove the left-over ethanol or toluene. The dewaxed sample (10g for each sample) was then subjected for delignification using microwave-assisted acidified sodium chlorite method. Two microwave power levels (640 and 800 W), three concentrations of sodium chlorite solution (0.4, 1.7, and 3.0 %), and three microwave treatment times (4, 8, and 12 min) were used in the experiment. The pH of the sodium chlorite solution was adjusted to 3.9 using 5% glacial acetic acid solution. A single charge of sodium chlorite was used in a particular experiment. Immediately after treating the sample (in the solution of acidified sodium chlorite) by microwaves for a particular duration, the reaction was quenched using a copious amount of ice-cold distilled water. The solution was repeated filtered (using Whatman Glass Fibre filter, 934-AH, Cat No. 1827047) and washed with distilled until the filtrate became pH neutral. The delignified biomass obtained was then carefully removed and dried until constant weight. The weight of each sample was recorded and the amount of lignin removed was obtained by the weight difference method as given by Equation 1.1. The schematic representation of the various steps in the experiment is depicted in Figure.1

$$\%\,Delignification = \frac{M_i - M_f}{L_i} \times 100 \qquad \textbf{1.1}$$

Where,

M_i and M_f - Initial and final mass of the sample (before and after delignification)

L_i - Amount of lignin initially present in the sample

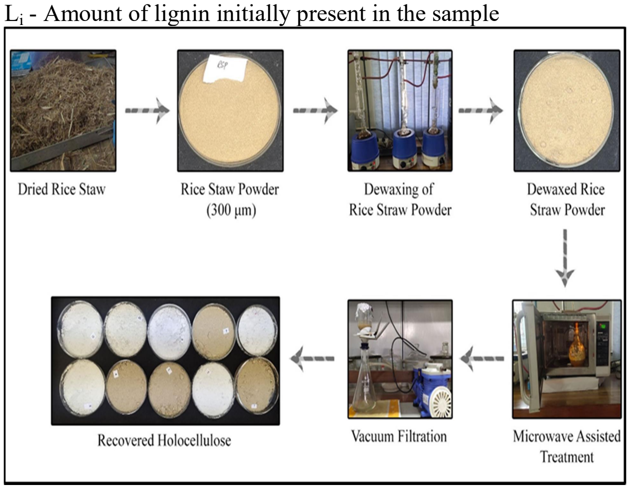

Figure 1: Schematic flowchart of the experimental procedure

Results and Discussion

The present study aimed to reduce the time required for delignification of rice straw biomass and also enhance the effectiveness of cleaving the lignin bonding using a microwave-assisted approach. Different experimental conditions were tested in the study. The initial composition of the rice straw revealed the results shown in Table 1.The results are congruent with those reported in previous literature (Cheng *et al.*, 2011; Dutta *et al.*, 2014; Yadav *et al.*, 2016). Variations in the initial composition pertained to the difference in variety, geographical conditions and cultivars. After subjecting the dewaxed rice straw powder to microwave treatment, the colour of the samples changed from brownish to light brownish and white (for different experimental conditions) as shown in Figure 2. The change of colour signifies the removal of lignin from the dewaxed rice straw powder. At lower concentrations of sodium chlorite solution (0.4%), the colour changed to light brown, which signified the presence of residual lignin, while in the case of other concentrations (1.7 and 3.0 %) the colour was off-white and white respectively.

The delignification percentage (DP) achieved after the microwave treatment at different experimental conditions is shown in Table 2. The DP of rice straw biomass in the present study varied from 24.7 to 90.12%. The results signified that the application of microwave-assisted method was successful in delignifying the samples effectively and quickly. The graphs of the results at two microwave powers are shown in Figures 3 and 4.

Table 1. Initial Composition of the rice straw

S. No	Composition	Average Value (%)
1.	Moisture Content	9.99
2.	Extractives	7.51
3.	Acid Soluble Lignin	3.86
4.	Acid Insoluble Lignin (Klason-Lignin)	10.83
5.	Cellulose	34.31
6.	Hemicellulose	31.70

Table 2. Delignification percentage at different experimental conditions

Microwave Power (Watt)	Irradiation Time (Minutes)	Percent Delignification (%) (Based on initial amount of lignin)		
		Bleaching Solution Concentration		
		C_1(0.4%)	C_2 (1.7%)	C_3 (3.0%)
800	4 Min	26.7	56.43	78.76
	8 Min	26.9	59.32	85.91
	12 Min	27.5	63.11	90.12
640	4 Min	24.7	50.12	66.54
	8 Min	25.9	52.33	74.21
	12 Min	26.5	60.17	88.21

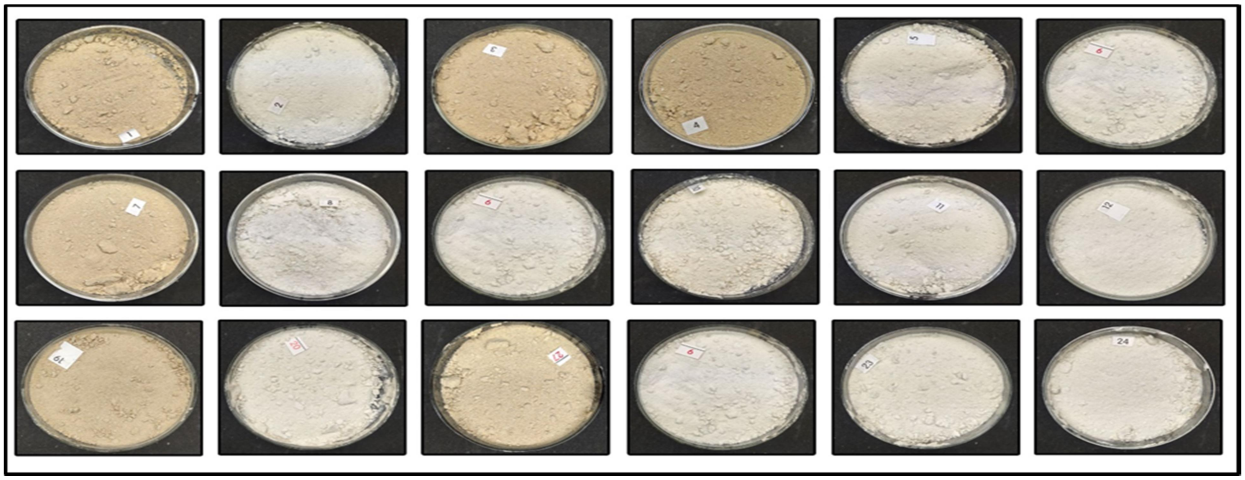

Figure 2: Differential change in colour of dewaxed rice straw powder after microwave assisted chemical treatment at different conditions

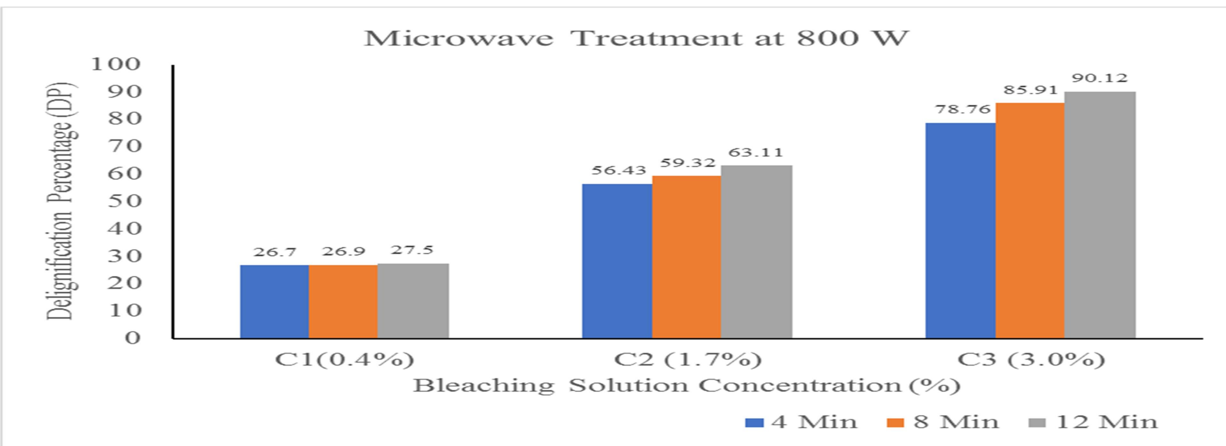

Figure 3: Delignification Percentage at Microwave Treatment of 800 W

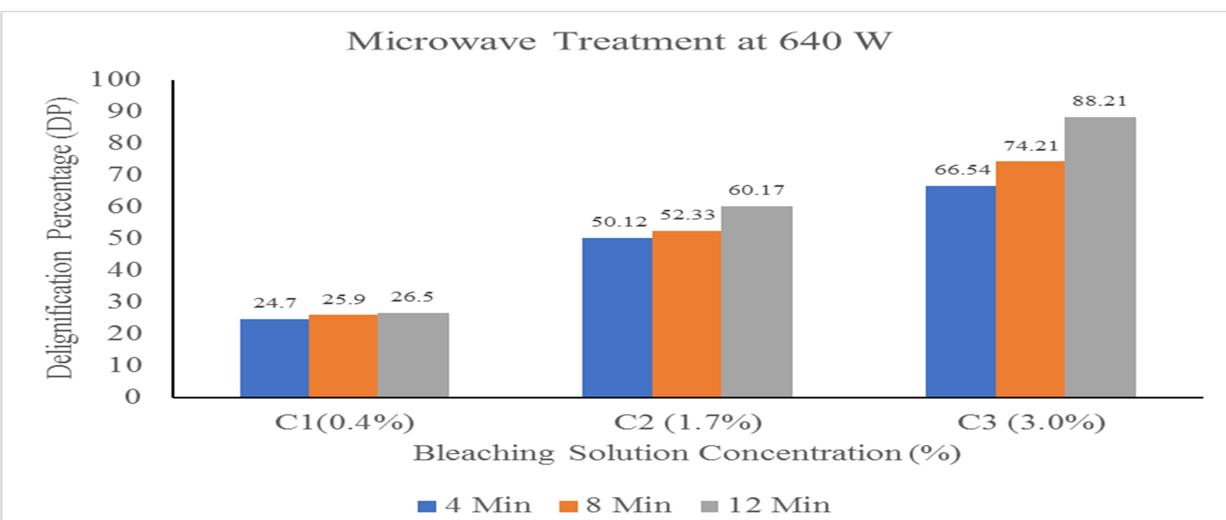

Figure 4: Delignification Percentage at Microwave Treatment of 640 W

Effect on Microwave power, Irradiation time and Bleaching Solution Concentration on the Delignification of Rice Straw

The effect of microwave power on the delignification can be seen from figures 2 and 3. At 640 W, the delignification percentage varied from 24.7 to 88.21% for different concentrations and irradiation times, while at 800 W, the delignification percentage varied from 26.7 to 90.12%. The delignification percentage achieved in this study was comparable and even better than the results obtained for delignification of various lignocellulosic biomasses like red meranti woodsaw dust (97%), maize straw (78.0%), rye straw (68.8%), rice straw (82.1%) using conventional sodium chlorite and other bleaching methods for long durations with multiple charges (Noredyani *et al.*, 2020; Xiao *et al.*, 2001). (Kumar *et al.*, 2013) achieved delignification of 90-95% using conventional sodium chlorite bleaching (with three charges of sodium chlorite) method in switchgrass, poplar, corn stover, and pine sawdust. Regarding microwave-assisted treatment, 40 and 80% delignification was achieved by microwave-assisted water and peracetic acid treatment of pineapple waste (Azelee *et al.*, 2019). The results confirmed the disruption of lignin-carbohydrate complex and effective delignification due to application of microwaves. Also, delignification of rice husk by microwave-assisted treatment at 240 watts resulted in almost 30% delignification in just 5 minutes (Laghari *et al.*, 2018). The effect of microwaves is due to the enhanced cell wall rupture and subsequent diffusion caused by unidirectional inside out heat and mass transfers (Veggi *et al.*, 2013). On increasing the microwave power from 640 to 800 W the increase in delignification percentage was achieved more at higher concentrations and less at lower concentrations as seen in figures 2 and 3. Similar results were reported by (Azelee *et al.*, 2019) where optimized microwave-assisted water and peracetic acid delignification was achieved at 750 and 550 watts respectively. The results showed that on increasing microwave power, the delignification increased in both cases, an optimum of 550 W and 750 W for water and peracetic acid respectively, which confirms the results of this study. The change in concentration had a profound effect on the delignification percentage of rice straw. On increasing the concentration from 0.4% to 3.0%, the delignification percentage increased from 24.7 to 88.21 % and 26.7 to 90.12% for microwave powers of 640 and 800 W respectively. Conventional sodium chlorite bleaching of lignocellulosic biomass has shown same or lower delignification percentages but with higher concentration and number of charges of sodium chlorite. The difference is because the availability of lignin for removal is more due to the application of microwaves which destroys the structure and makes the lignin of both the lumen and middle lamella available for the bleaching solution (Siqueira *et al.*, 2013). Delignification percentage of about 97% was achieved for red meranti sawwood powder but at a concentration of 25% sodium chlorite using the conventional water bath heating method (Noredyani *et al.*, 2020). Park *et al.* (2015) delignified mixed hardwood chip powder through conventional hot water bath bleaching using 41% sodium chlorite with a percentage delignification of about 77% albeit addition of five to six charges each hour. Laghari *et al.* (2018) found that on subjecting rice husk to microwave-assisted chemical treatment (with sodium hydroxide, sodium carbonate, hydrogen peroxide and sulfuric acid) the delignification percentage increased by increasing the concentration from 2 to 5%, except for sodium hydroxide where the maximum delignification of about 43% was seen at 3.5% concentration and this being highest of all the treatments. Thus, in the present study, a clear reflection of a synergistic effect on the increase in delignification percentage due to application microwaves and increased concentration of sodium chlorite is seen. Due to the application of microwaves, the time of delignification has significantly reduced to 12 minutes as compared to the time (almost 6h or more) required for the same or less percentage of delignification at higher concentration in conventional sodium chlorite bleaching process (Jin *et al.*, 2019; Kohli *et al.*, 2020; Kucharska *et al.*, 2018; Kumar *et al.*, 2013). Subhedar and Gogate (2014) delignified newspaper waste using sodium hydroxide and got a delignification percentage of about 80.0% and 40.2% with and without the use of ultrasound at a time duration of 70 min and 6h respectively. Thus, the use of microwave-assisted chemical treatment greatly reduces the time of the bleaching process.

Similar results for reduction in bleaching time (up to 10-15min) using microwave-assisted treatment has been reported in literature (Azelee *et al.*, 2019). The time reduction for bleaching using microwave-assisted alkaline, organosolv, sodium chlorite, ionic liquid and acetosolv treatment has been reported in literature (Akhtar *et al.*, 2014; Avelino *et al.*, 2019; Shengdong *et al.*, 2005; Sun *et al.*, 2019; Yaakob *et al.*, 2020). The effect of varying microwave irradiation time or treatment time also has a significant effect on variation of delignification percentage. However, the difference at the lower concentrations is not highly significant as compared to the higher concentration at both the microwave powers. At 640W, the delignification percentage change was about 1.8% in going from 4 to 12 min as compared to only 0.8% for same time increase for 800W. Similar pattern can be seen at higher sodium chlorite solution concentrations of 1.7 and 3.0%. This is probably because, there is a gradual lignin-carbohydrate complex breakdown at lower microwave power than at higher microwave power which results in quick consumption of lignin at higher microwave power with little difference or changes with time at the same concentration as compared to that at lower microwave power.

The microstructure of the rice straw powder and the delignified rice straw sample (with the highest delignification percentage of 90.12%) was evaluated through the scanning electron microscope (SEM). The SEM images of both the rice straw powder and the delignified sample are shown in Figure 5 (a & b). It is quite clear from the images, that, initially (Fig.5a) the surface of the rice straw powder is packed and smooth, which is due to the closed and complex structure formed by lignin with cellulose and hemicellulose. However, on delignification (fig. 5b), due to the lignin-carbohydrate cleavage and removal of lignin a more porous and rough structure can be seen.

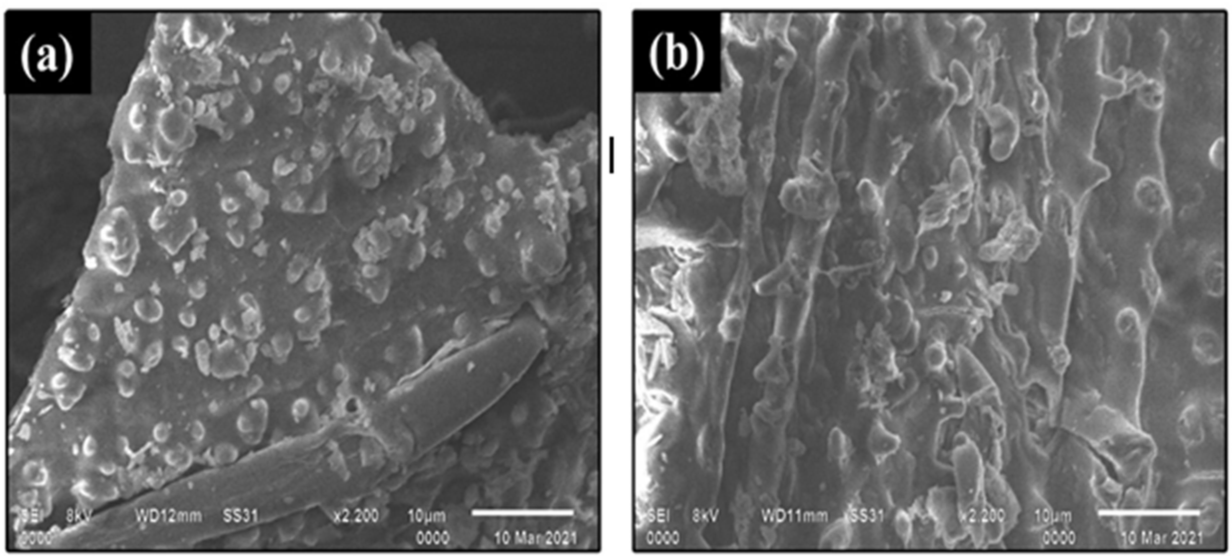

Figure 5. Scanning electron microscope image **(a)** Rice straw powder **(b)** Delignified rice straw powder

Conclusion

In the present study, Microwave-assisted chemical treatment of rice straw led to its quick and effective delignification. The quick and intense delignification of the rice straw with a single charge of sodium chlorite (as compared to multiple charges used in conventional sodium chlorite delignification) directly reduces the energy use along with the amount of chlorine generated in the process. This reduces the impact on the environment and subsequently the carbon footprint. Besides this, both the aforementioned facts have the potential of cost reduction in the scale-up mode.

The experiments conducted revealed that the delignification percentage varied from 24.7 to 90.12% for the experimental conditions. The maximum delignification percentage was obtained at microwave power, irradiation time and bleaching solution concentration of 800W, 12 min and 3.0% respectively. Moreover, the microstructure of the delignified sample revealed the disruption of lignin-carbohydrate complex and removal of lignin. The process can further be taken for optimization using a wider parameter range and in-depth characterization of delignified samples could be done.

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