

Enhancement of Ethanol Separation from Aqueous Solution by Adsorption Technique

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Abstract— Because of the huge industrial development and the high dependency on fossil fuel leading to high air pollution which causes a variety diseases such as respiratory diseases and disaster of global warming all these severe results encourage the researchers to figure out an alternative fuel which has a low environmental impact, the promised one is the bioethanol. The basic process to produce bioethanol is the fermentation for biomass, the most challenging of this process is the separation of bioethanol. This work concerns with modification of bioethanol separation from simulation aqueous solution of the fermentation by adsorption method. The study assesses different types of the feedstock of the fermentation as adsorbents based on the selectivity and capacity. The capacity values via water for yellow corn, wheat bran, rice straw and wheat straw were 0.24, 0.28, 0.15 and 0.14 respectively, and the selectivity values via water were 4, 2, 3, and 2.2 respectively. The first ethanol separation stage of adsorption (after the fermenter) used sepabeads207 to purify ethanol from 10 wt% to 50 wt% used in previous study. In this study, the second separation stage was established including crushed y-corn and wheat bran giving ethanol purity of ~60wt%. The third separation step used y-corn and rice straw to introduce a bioethanol ratio for ~70wt%.

Keywords— Adsorption, ethanol, crushed corn, wheat bran, rice straw, wheat straw.

I. I. INTRODUCTION

The global warming is a crucial and fatal ecumenical problem around the world [1]. This problem is related directly with emissions of fossil fuel combustions and other industrial gases such as CFC, hydrocarbons, CO₂ and other types of greenhouse gases [2]. The huge amounts of these effects on atmosphere and environment come from using fossil fuels (oil, gas and coke). The revolution of industry was built, essentially, on this type of fuel because of its availability in large quantities [3]. To occupy this disaster and resolve it (even partially), the researchers persevere to find an alternative fuel that can be used side by side with fossil fuels to decrease the dependency on it (one day will disappear) and decrease the excess of the CO₂ and other greenhouse gases in atmosphere [4].

Using renewable fuel leads to decreasing the problems of excess of CO₂ emissions, global warming and other environmentally damages by inserting the effluent in the closed cycle (combustion of biomass emits effluent to atmosphere and photosynthesis pulls this effluent to form biomass again and so on) [2]. One of reliable alternative fuel is the bioethanol, also named ethyl alcohol (C₂H₅OH).

It is a colorless liquid, biodegradable, low in toxicity, non-smoke flame and produces little pollution to the environment because it has a high ratio of H/C [5], which is produced by bioprocess depending on biomass. It is used as fuel for gasoline engines that have already been built (may need to be fabricated) and also as fuel oxygenated to enhance the octane number of gasoline with low quality by mixing a certain ratio to produce a blind (such as E85, ethanol=85, gasoline=15) [6], also the bioethanol can be used for combustion in different applications because it burns in clean flame without smoke [7].

Alternative fuel should be sustainable and economical to be successful in replacing traditional fuel [8]. There are different ways to produce sustainable fuel based on biomass such as direct combustion, pyrolysis, chemical conversion, biological conversion [2].

The promised way to produce bioethanol is the fermentation process (classified under biological conversion) [9]. The fermentation process depends upon the biomass as a renewable source. The bioethanol which produced from the fermentation process has purity with about 10wt% (based on the microorganism and the fermented substrate) [10]. The challenge of producing bioethanol as a fuel is the purification process.

There are many techniques for separating bioethanol from an aqueous solution (fermentation's broth) and purify it. Classical distillation [11], azeotropic distillation [12], extractive distillation with salts [13-14], pervaporation [15], [16] and adsorption [17].

All distillation processes are effective processes for getting high purity for bioethanol (~95wt%) but, they are suitable for high concentration of ethanol (start with high concentration e.g. 70%) and a large amount, the



disadvantage of the distillation technique is needing a lot of heat to evaporate the mixture and this lead to consume high energy i.e. high cost, so these are not suitable for outlet bioethanol with purity 10wt%.

The other separation technique is pervaporation; this method needs expensive equipment to compress the solution through selective costly membrane to separate bioethanol and because of the fermentation medium includes many suspended solids it makes fouling for the membrane, Table 1 summarizes the techniques of bioethanol separation and their advantage and disadvantages.

For the low purity of bioethanol produced by the fermentation operation, a suitable technique should be selected. A chosen technique must give suitable purity for ethanol as a fuel (or for other applications), high yield and economic price. Adsorption is one technique that gives a good result in this field and in water treatment techniques [18] and [19]. The preference of this method is excluding adding heat (only little amount to maintain the temperature as the best value) as compared with distillation. Adsorption technique has many advantages such as using the waste of agriculture products, low cost because no need to use heat, can be combined with a unit of bioethanol production and the adsorbents can be reused in the same unit or in another process [17].

Table 1 explains the separations techniques and their advantage and disadvantages [17]

Process	Conditions	Percentage of purity	Advantages	Disadvantages
Adsorption(ethanol) by silica or suitable adsorbent	Cons. (T & P) Using 4 or 2 Columns	95 %	Low cost, high purity	Semi Batch process
Ordinary distillation	T=77C, P=1 atm 1 Column	92 %	Continues process, ethanol without impurities	Expensive, high-energy consumption
Azeotropic distillation	Adding entrainer 3 columns	99 %	Continues process, ethanol without impurities	Expensive depend on entrainer
Liquid-liquid extraction	Suitable extractor	During production	Low energy consumption Increase production of fermentation	Difficult in choosing suitable extractor and Stripper

Adsorption phenomenon depends on the variation in molecular size and dipole momentum between ethanol and water [20-21]. Also, it depends on active forces within the boundaries between the surface of the adsorbent and the molecules in the solution [22]. The efficiency of the adsorbent is based on two factors: first, the number of vacancies inside the mass and their dimensions and suitability with dimensions (or size) of molecule type. Second, the chemical-physical forces of active sites inside the pores. The Number of vacancies decides the capacity of the adsorbent and the power of the forces decides the selectivity of it [23-25]. Some adsorbents selective to water and some are selective to ethanol. In this case, produced bioethanol with a high percentage of water, the first stage of separation is better to use either an adsorbent with high selectivity of bioethanol or an adsorbent with high capacity (even low in selectivity but larger than 1) of water to remove a large amount of water. In the previous study [26],

ethanol was concentrated from 10wt% to 50wt.% using a single stage of adsorption with adsorbent sepabeads207. This study includes testing other materials as adsorbents which give high purity and quantity of ethanol. In the discussion part, the materials were tested are: crushed yellow corn, rice straw, wheat straw and wheat bran. These materials were elected because they are used as fermentation precursor.

The modernity of this study is using the feedstock materials of the fermenter as separators agent to save the ethanol and water (medium of the fermentation) without losing and decrease the final cost of bioethanol by non-using external adsorbent. In addition to evaluate the ability of this material (yellow corn, wheat bran, rice straw and wheat straw) to adsorb water and ethanol because there is very little information about this role.

II. CHARACTERISTIC OF MATERIALS:

The materials that tested were: crushed corn, wheat bran, rice straw and wheat straw.

The selection of materials to be adsorbents was done based on availability in the region, low cost because they are waste of agriculture process (no need to be regenerated), also, these materials are used as a precursor to feeding the fermenter.

There are some properties of materials govern using them as adsorbents such as:

- Capacity (of water): is define as the ability of adsorbent material to include water, referred as C [27].

$$C = \frac{\text{wt.of water on adsorbent}}{\text{wt.of adsorbent}} \quad [27]$$

- Selectivity (of water): is define as the ability of adsorbent material to select water to adsorb it more than ethanol or other contents, referred as S [27].

$$S = \frac{\text{water wt ratio in adsorbent}}{\text{water wt ratio in origin solution}} \quad [27]$$

$$S = \frac{\left(\frac{\text{gm of water}}{\text{gm of ethanol}}\right)_{\text{in adsorbent}}}{\left(\frac{\text{gm of water}}{\text{gm of ethanol}}\right)_{\text{in origin solution}}}$$

These materials used as adsorbent in second stage because the first stage was done by using sepabeads207 and get purity of bioethanol about 50wt%.

III. THE PROCEDURE

The materials were grinding and sieved about 1000 - 2000 μm .

Four beakers (and 4 replicate) of solution of ethanol were prepared with concentration of 50wt% and volume 50 ml for each one.

- Preparation of ethanol solution with 50wt% and volume 400 ml:

$$\begin{aligned} \rho_e &: \text{ethanol density at } 20^\circ\text{C} \\ &= 788.886 \text{ kg} / \text{m}^3 \\ \rho_w &: \text{water density at } 20^\circ\text{C} \end{aligned}$$

$$= 1002.09 \text{ kg} / \text{m}^3 [5]$$

V_e : ethanol volume

$$\text{Wt. \%} = \frac{\text{wt.of ethanol}}{\text{wt.of ethanol} + \text{wt.of water}} 100\%$$

$$0.5 = \frac{\rho_e * V_e}{\rho_e * V_e + \rho_w * V_w}$$

$$0.5 = \frac{\rho_e * V_e}{\rho_e * V_e + \rho_w (400 - V_e)}$$

$$\rho_e * V_e = 0.5 * \rho_e * V_e + 200 \rho_w - 0.5 V_e \cdot \rho_w$$

$$\rho_e * V_e - 0.5 \rho_e \cdot V_e + 0.5 V_e \cdot \rho_w = 200 \rho_w$$

$$V_e (\rho_e - 0.5 \rho_e + 0.5 \rho_w) = 200 \rho_w$$

$$V_e = \frac{200 \rho_w}{0.5 \rho_e + 0.5 \rho_w} = \frac{200 * 1002.09}{0.5(788.886 + 1009.09)}$$

$V_e = 223.8 \text{ ml}$, volume of ethanol mixed with 176.2 ml of water to produce 400 ml of 50wt% of ethanol solution.

The tested materials were weighed and fixed at 10 gm for certain kinds. Each type of material was put in the beaker including the solution of ethanol. The conditions of the adsorption process are: soft mixing was applied for content of each beaker with a fixed time (10 min.), the temperature stayed constant (about 5 °C), which is the temperature of the solution coming from the first adsorption stage. Stripping process was utilized to get the adsorbed solution. Each solution including solid materials was filtered and removed free solution from solid materials then, the adsorbent was transferred to a glass column with two permitted cups (to keep the adsorbent inside the column and allow the solution to pass through it) and covered by a jacket (to control temperature). A hot air (temperature = 80 °C and Time=15 min.) passed inside the column through the adsorbent to evaporate the adsorbate and condensate it by condenser using cold water (with temperature = 5 °C) as in Fig. 1, then the condensation was collected and measured its volume and analyzed by GC-MS to get ethanol purity.

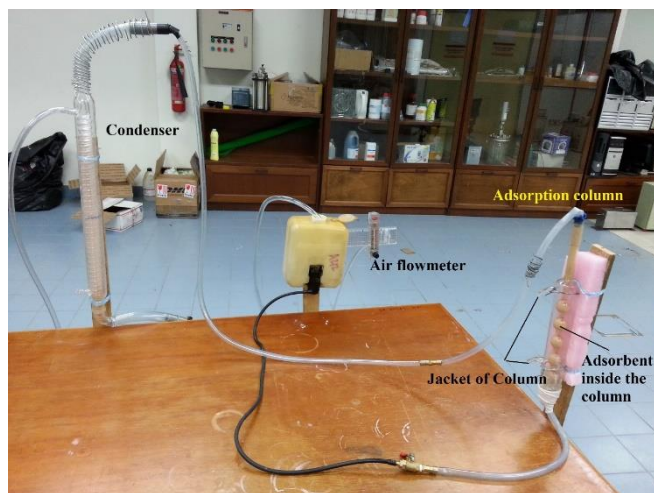


Fig. 1: Stripping set of adsorbate solution

IV. RESULTS AND DISCUSSION

There are important definitions that should be explained: Adsorption: is a common phenomenon that occurs at the time that a gas or liquid (fluid) is in contact with a solid. The molecules of the fluid are retained by the superficial atoms of the solid and inside the pores (if there) and concentrated at the solid surface.

Desorption: or called stripping is a revers phenomenon of adsorption which refers to leaving of attached molecules the solid surface towards the fluid bulk.

Adsorbent: is a solid material which has a property for surface molecules to interact with some fluid molecules.

Adsorptive: is the fluid (still in bulk phase) that has characteristic to be adsorbed on a specific solid surface.

Adsorbate: the fluid molecules virtually are adsorbed on the solid surface [28].

Fig. 2 shows the adsorbent, adsorbate and the pores [29].

V. THE SELECTIVITY AND CAPACITY

The selectivity and capacity are highly important parameters, the materials were tested to adsorb water (water selectivity and capacity) from ethanol – water solution under constant conditions, temperature 5 °C, soft mixing with time 10 min., the results are illustrated in Table 2.

Crushed y-corn has high capacity (0.24 g/g), which means it has a high percent of vacancies inside these materials, high internal surface area and the chemical structure includes hydrophilic groups (such as starch which is hydrophobic), these characteristics give the adsorbent capability to occupy amount of water reach to 20% of their weight. The value of selectivity for crushed y-corn is the highest (4) (according to tested materials). The justification of this property is the pores dimensions and caves inside the material are suitable for the volume of the water molecule and it is not for ethanol molecule, the volume of the water molecule is (29.7 Å³) [30].

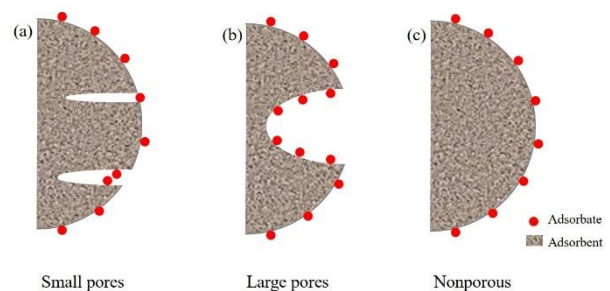


Fig. 2: Explains these definitions

Table 2 the selectivity and capacity of tested materials under adsorption conditions

	Material			
	Crushed y- corn	Wheat bran	Rice straw	Wheat straw
wt. of water, g	2.4	2.8	1.5	1.4
wt. of ethanol, g	0.6	1.4	0.5	0.636
Capacity via water, g/g	0.24	0.28	0.15	0.14
Selectivity via water, g/g	4	2	3	2.2

while the ethanol molecular volume is (97.27 Å³) [30] as in Fig. 3, in addition to the nature of adsorbent molecules and the chemical structure of the corn has a lot of hydrophilic groups [19], [26] as in Fig. 4A.

Wheat bran has the highest capacity (0.28 g/g). This value is related to the same reasons of y-corn but, for the selectivity it has the lowest value (2), this means the dimensions of pores are big enough to retain both types of molecules ethanol and water in spite of the difference in sizes as in Fig.3 and 4B.

Rice straw has low capacity (0.15g/g) and good selectivity (3). The low capacity means low numbers of caves and the good selectivity means the type of the pores is very suitable for water molecules dimensions and not allow ethanol molecules to settle in these pores as in Fig. 3 and 4A.

Wheat straw has a capacity of (0.14 g/g) premised on the high content of lignin and cellulose which include a low percent of caves inside their structure, so there is limited space to hold water molecules. Also, it has low selectivity (2.2) means the vacancies are limited and are not selective for water only but can receive water and ethanol molecules for a little privilege for water molecules.

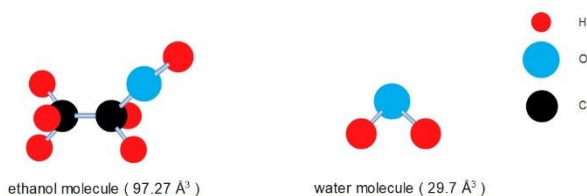


Fig. 3: The difference between molecular volumes of ethanol and water

Fig. 4 explains the difference between high and low selectivity adsorbent. The material A has pore size suitable for water but is not suitable for ethanol so, the vacancies are occupied with water only and the ethanol was adsorbed only on the surface, the ratio of water molecules/ethanol molecules over adsorbent is high.

The material B has pore size is large via both molecules (water and ethanol) so, the caves are taken by two types of molecules thus, the ratio of water molecules/ethanol molecules is low (still > 1).

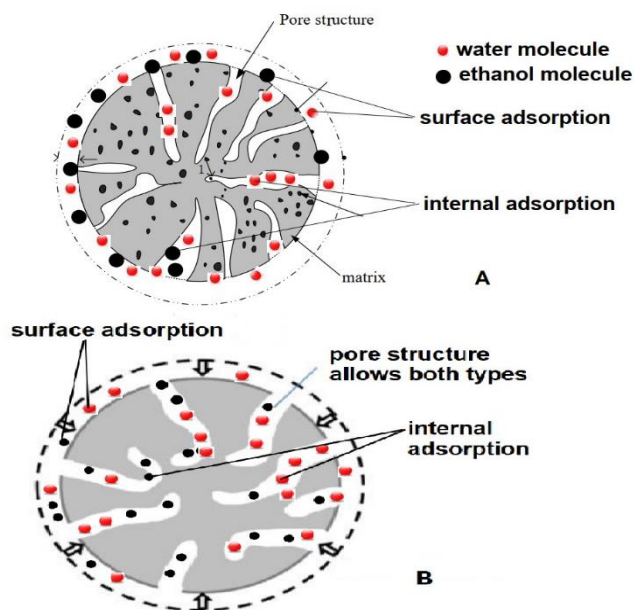


Fig. 4: A high selectivity, B low selectivity adsorbent

The governor of the selectivity for the adsorbent towards a certain adsorbate, two conditions should be available: the size of pores and the nature of function groups of internal surfaces [26].

The advantage of determination of the capacity and selectivity for each material to use them in proper sequence of the separation stages.

The material with high capacity via water (selectivity via water >1) should be used in the purifying stage when the solution consists of water in high ratio, in this case must use adsorbent with high capacity of water to remove major of water with losing small amount of ethanol (will return to the fermenter as a precursor).

The adsorbents with high selectivity via water (even with low capacity) are recommended to be used in the step of separation when the solution consist of ethanol in high ratio, should use the adsorbent with high selectivity of water to rise the concentration of bioethanol, for appropriate design the material has to remove water more than ethanol.

In the case of this work, the first stage of ethanol separation (after the fermenter) used in previous study using sepabeads207 to increase bioethanol ratio coming from the fermentation process with 10wt% to ~50wt% under adsorption conditions: Time = 5 min., T = 20°C, pH = 4, and the stripping of the adsorbate from adsorbent with conditions of Time= 15 min., T= 80°C (for evaporation), T= 5 °C (for condensation) [26]

. In the second purifying stage, should use the adsorbent of high capacity of water, selectivity should be more than 1, (crushed corn and wheat bran) because these materials (as adsorbent) no need to be regenerated, since it needs to be fed to the fermenter with water content as in Fig. 5. In this stage, the concentration of bioethanol became 58 – 60wt%, as in Table 3.

Table 3 the bioethanol purity after using crushed corn & wheat bran

Adsorbent Material	Bioethanol concentration wt% input	Bioethanol concentration wt% output	Adsorption conditions
Crushed corn & Wheat bran	50%	58-60wt%	T= 5 °C Time=10 min.

In the third separation stage, the used adsorbent must be with high selectivity of water (crushed y-corn and rice straw) to increase concentration of bioethanol. Also, the adsorbent can be fed to the fermenter as in Fig. 5. By the third stage, the concentration of bioethanol reaches about 69-70wt% as in Table 4.

Unfortunately, there is no abundant information about using these materials (yellow corn, wheat bran, rice straw and wheat straw) as adsorbent for ethanol and water to compare the result of this work with results from other literature, the information scarcity in this field gives this study a privilege to introduce absent information can be used to establish an application based on it.

Table 4 bioethanol ratio after using crushed corn & rice straw

Adsorbent Material	Bioethanol ratio wt% input	Bioethanol ratio wt% output	Adsorption conditions
Crushed corn & Rice straw	~58wt%	~70wt%	T= 5 °C Time=10 min.

VI. UNIT OF ETHANOL PRODUCTION

The fermenter is a bioreactor that convert bio mass to ethanol with percent ~10wt%. The outlet of the fermenter goes to first separation stage using sepabeads207. This stage includes two adsorption equipment, A in the adsorption process and the B in the stripping process (desorption for solution from adsorbent), when the material in A equipment becomes saturated, the process will be reversed, B in the adsorption process and A in regeneration. The conditions of adsorption process for first stage as determined by [17] are: temperature= 20 °C, pH= 4 and time = 5 min. and the conditions of the stripping process as mentioned by [17] are: temperature = 80 °C and time= 15 min. and the vapor is condensed with T=5 °C. The solution of bioethanol coming out from stripping process is in ratio of ~50wt%, this solution enters a second separation stage using crushed y-corn and wheat bran is purified (by adsorption of water) to about ~60wt% at conditions of T = 5 °C and time= 10 min. and the adsorbents (y-corn and wheat bran) enters to the fermenter as precursors (no need to remove adsorbed solution on them) as in Fig. 5. The bioethanol solution goes out from the second stage steps inside the third separation stage which use y-corn and rice straw as adsorbent. The solution goes out of the third stage and has a purity about ~70wt% and the saturated adsorbents enter the fermenter as substrate, also no need to be regenerated as in Fig. 5.

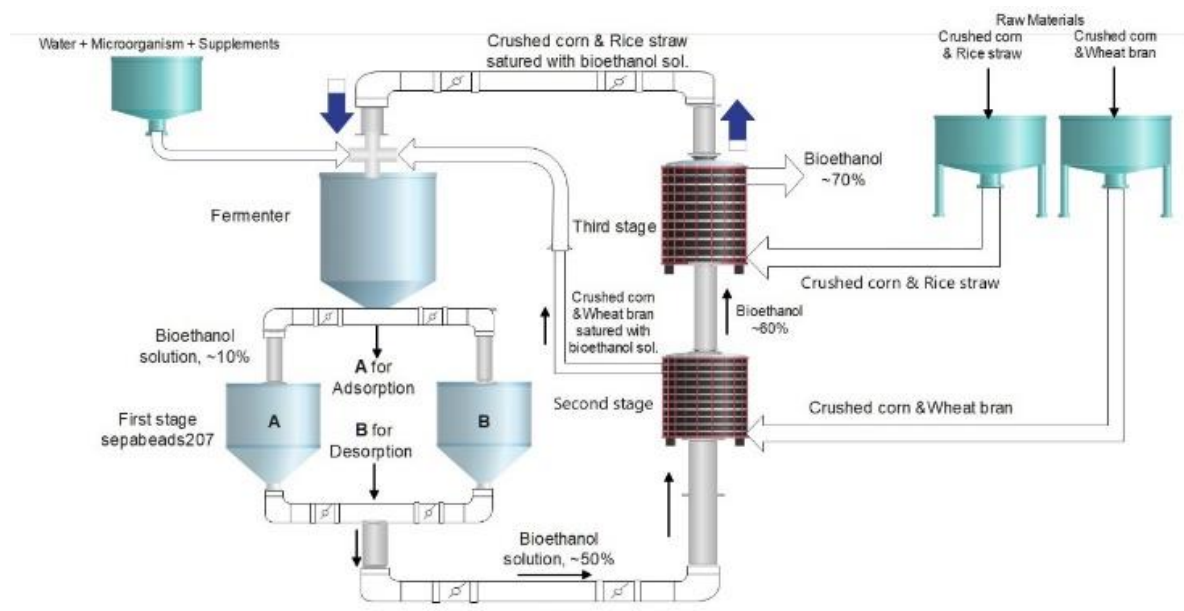


Fig. 5: Process diagram for the fermentation and adsorption processes

The novelty of this study is rising the bioethanol concentration from 50wt% to ~70wt% without using external adsorbent but, using the precursors, themselves, of the fermentation as adsorbents for outlet product as in diagram in Fig. 5.

VII. CONCLUSION

Some of the waste of agriculture such as y-corn, wheat bran, rice straw and wheat straw are used as a feed for the fermenter. This work aims to assess of capability of using

these materials as adsorbent to rise the concentration of bioethanol without additional cost. The results show the capacity via water for crushed y-corn, wheat bran, rice straw and wheat straw are 0.24, 0.28, 0.15 and 0.14 respectively and the selectivity via water are 4, 2, 3 and 2.2 respectively. Using crushed y-corn and wheat bran in the second separation stage raised the bioethanol concentration from ~50wt% to ~59wt%. Utilizing crushed y-corn and rice straw in the third purification stage increased the bioethanol ratio from ~59wt% to ~70wt%. Using the precursor of the fermentation process as adsorbent installed to be a purifier for the bioethanol and introduce a reliable purity without using external (costly) adsorbents.

CONFLICT OF INTEREST

Authors declare that they have no conflict of interest.

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