CPE-79

Modelling and Optimisation of *Eurycoma Longifolia* Extraction Utilising a Recirculating Flow Extractor

Mohd Ajib Mohtar[1] **Sivakumar Kumaresan**[2*] **Mohd Roji Sarmidi**[2] **Ramlan Abdul Aziz**[2]

[1]*Chemical Engineering Department Faculty of Chemical and Natural Resource Engineering*
Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, Malaysia
Tel: +60-12-7421296, E-mail:mohd_ajib83@yahoo.com

[2]*Chemical Engineering Pilot Plant*
Universiti Teknologi Malaysia, 81310 81310 UTM Skudai, Johor, Malaysia
Tel: +60-7-5531662 Fax: +60-7-5569706,
E-mail:shiva@cepp.utm.my, mroji@cepp.utm.my, ramlan@cepp.utm.my

Abstract

In this study, Tongkat Ali was extracted with a newly designed recirculating flow extractor with temperature and flowrate as the operating parameters. A preliminary study determined the optimum duration and ratio for extraction to be 90 minutes and 40:1 w/w. The determination of optimal operating parameter value for this extractor was based on maximum percentage extract yield and solid diffusivity, D_s, value. From the experiments, it was found that the temperature and flow rate that produce the highest yield and solid diffusivity value are at 90°C and 400 rpm (22.47 ml/s). The optimal operating parameter values were used to compare the recirculating flow extractor performance with a batch extraction at 90 °C. The comparison showed that the batch extraction was able to extract more rapidly than the recirculating flow extractor. The solid diffusivity, D_s, value for the batch extraction was found to be is $3.12 \times 10^{-11}\ m^2s^{-1}$ while the recirculating flow extractor had a solid diffusivity, D_s, value of $2.98 \times 10^{-11}\ m^2s^{-1}$ which indicated the difference in extraction rate. However, the extractions utilizing the recirculating flow extractor produced a higher final yield than batch extraction at 7.70 % (w/w) for the recirculating flow extractor and 6.67 % (w/w) for the batch extraction. This is possibly caused by the higher rates of solvent losses through evaporation for batch extraction.

Keywords: *Eurycoma longifolia*, phytochemical processing, herbal processing, modelling and optimisation

Introduction

Eurycoma longifolia, commonly known as Tongkat Ali, is native to the jungles of Malaysia as a plant known to have traditional medical applications [1,2]. In Sumatra it is grown as a commercial plant in a 20 hectare plantation to supply the manufacturing production and save the plant from extinction [3].

Traditionally, Malaysians have used Tongkat Ali to treat several diseases such as cancer, malaria, fever, skin diseases, diabetes, and ulcer. It is also used as tonic after child birth. It has became known worldwide recently due to its ability to treat erectile dysfunction and improve sexual desire.

The Malaysian Agriculture Research and Development Institute (MARDI) has reported that food supplements with the properties to enhance sexual health are becoming the fastest growing product within the herbal industry, and on the top of the list is Tongkat Ali [3]. Scientific research has shown that Tongkat Ali has properties related to antimalarial [4], anti-tumor promoting and anti-parasitic activities [5], and overcoming sexual dysfunctions [6]. These studies thus justifiy the benefit and miracle of Tongkat Ali to enhance human health.

Traditionally the roots of Tongkat Ali are boiled and the water is taken as a health tonic. However, due to its extremely bitter taste, many people do not prefer to drink the tonic. Batch solid liquid extraction and spray dryer technology has solved this problem where the Tongkat Ali extract is converted to powder to be put into capsule form. This processing method has been used to produce pure extract which is easy to handle and free of harmful bacteria and fungi. Based on its traditional uses and scientifically researched findings as stated above, Tongkat Ali extract has a high market demand. However, the current processing method based on batch solid liquid extraction has a low yield of 2-3% w/w. For this reason an alternative method of extraction utilizing a recirculating flow extractor was explored to maximize the yield of Tongkat Ali extracts.

Approach and Methods

The experimental work of this study was carried out to study the effects of temperature and flow rate on yield to find the optimal condition of extraction utilizing a recirculating flow extractor. A preliminary study was initially done to find the optimal time and ratio for further work in the main experiments. The range of extraction temperature was between 50°C to 90°C and flow rate is between 6.91 ml/min (100 rpm) to 32.85 ml/min (600 rpm) while the particle size of Tongkat Ali was fixed between 0.5-1.0 mm. No presoaking was done before extraction in the recirculating flow extractor. The weight of the Tongkat Ali sample loaded was 10 g.

Recirculating Flow Extractor

A recirculating flow extractor vessel and apparatus were designed and constructed as shown in Figure 1 and Figure 2. The extraction vessel was modified from a sampling bottle, where the bottom of the bottle was cut to install the filter. The filter used is made from a simple water tap filter. Then the cap and bottom were drilled to install the flow pipe. These flow pipes were taken from motorcycle tube components. The final work was to reconnect the bottom parts. The clip and rubber rope was used as the connecting material. The vessel dimension are 105 mm length with diameter 30 mm and be able to load 10-15 grams particle.

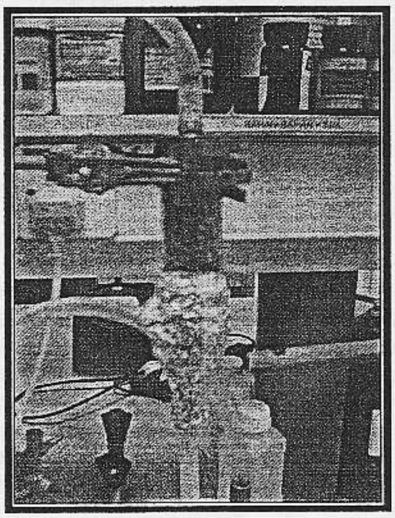

Figure 1: Extractor Vessel

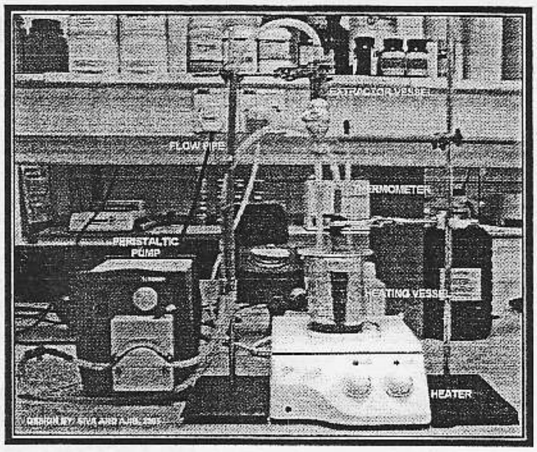

Figure 2: Recirculating Flow Extractor

Experimental Design for Temperature and Flow rate Studies

The experimental design was developed based on the chosen optimal duration and ratio to evaluate the effects of flow rate and temperature. The ranges of extraction temperature were between 50 to 90 °C. The solvent flow rates were of six flowrates ranging from 6.91 ml/min (100 rpm) to 32.85 ml/min (600 rpm). Revolutions per minute (rpm) is used as the reference value for flowrate as it is the control measurement for the peristaltic pump. The optimal liquid to solid weight ratio and extraction duration used was determined by the preliminary experiment.

Table 1: Experimental Design Index

Temperature (°C)	Flow rate (rpm)			
	100 (6.91 ml/s)	200 (12.09 ml/s)	400 (22.47 ml/s)	600 (32.85 ml/s)
50	A1	A2	A3	A4
60	B1	B2	B3	B4
70	C1	C2	C3	C4
80	D1	D2	D3	D4
90	E1	E2	E3	E4

Results and Discussion

Preliminary Time Study

From the extraction time study carried out as shown in Figure 3, the optimal maximum extraction time for a recirculating flow extractor was found to be 90 minutes above which continued extraction only resulted in minimal increase of concentration. This is slightly faster than a similar batch extraction study by Sim [2] which predicted an optimal maximum extraction time of 120 minutes for a batch process.

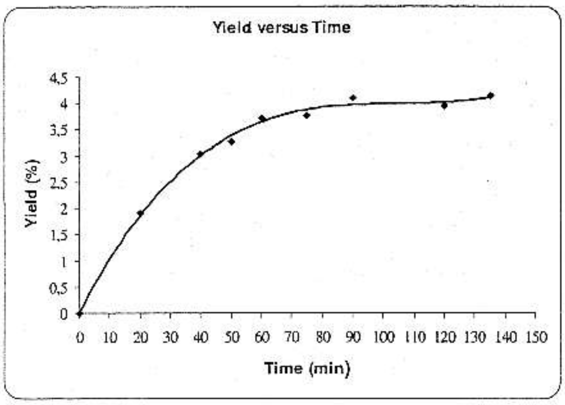

Figure 3: Percentage Extract Yield as a Function of Time

Table 2: Rate Constant, k_{obs} (s^{-1})

Flow rate (rpm)	Temperature (°C)				
	50	60	70	80	90
100 (6.91 ml/s)	7.78E-06	8.97E-06	9.03E-06	9.64E-06	9.7E-06
200 (12.09 ml/s)	8.78E-06	9.69E-06	9.94E-06	1.08E-05	1.15E-05
400 (22.47 ml/s)	8.89E-06	1.03E-05	1.08E-05	1.13E-05	1.17E-05
600 (32.85 ml/s)	8.75E-06	9.42E-06	9.87E-06	1.08E-05	1.08E-05

Preliminary Ratio Study

Preliminary ratio experiments were carried out to determine the optimal ratio to be utilized in the flow rate and temperature study against yield. The selection of ratio is based on weight ratio w/w in the range of 20:1 w/w to 50:1 w/w. The optimal ratio of the experiment was found at a ratio of 40:1 as shown in Figure 4 similar to the results by Kaur [7] for the batch process. From the data it is observed that the lower ratios produced higher final concentration whereas higher ratios gave lower final concentrations. The loss of solvent through evaporation is higher at the lower ratio and it is possible that this reduced the overall yield. Therefore, it is also due to this factor that the highest yields were produced at the higher ratio.

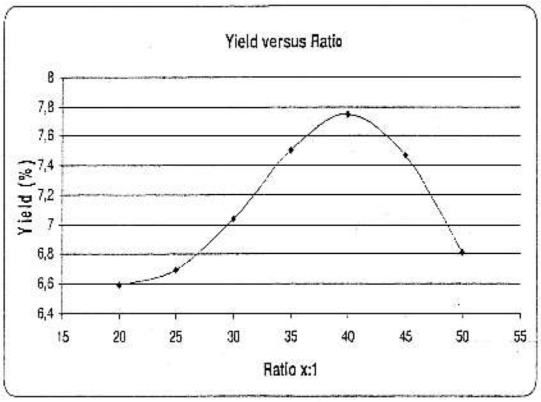

Figure 4: Percentage Extract Yield as a Function of Ratio

Rate constant, k_{obs}

Spiro and Jago's steady state kinetic model [8] which predicts first order behaviour was used to analyse the concentration data, which fitted an equation of the form:

$$\ln\left(\frac{C_\infty}{C_\infty - C_t}\right) = k_{obs}t \qquad (1)$$

where, k_{obs} is the first order rate constant (s^{-1}), C_∞ is the concentrations of solution at equilibrium (g ml^{-1}), C_t is the concentration solution at any time (g ml^{-1}) and t is the extraction time. Figure 5 shows one of the first order plots obtained. The value of rate constant, k_{obs}, can be obtained through the slope of the graph as done by Jaganyi and Price [9] in caffeine extraction. The rate constant , k_{obs}, values determined at different temperature and flow rate are summarized in Table 2.

Table 2: Rate Constant, k_{obs} (s^{-1})

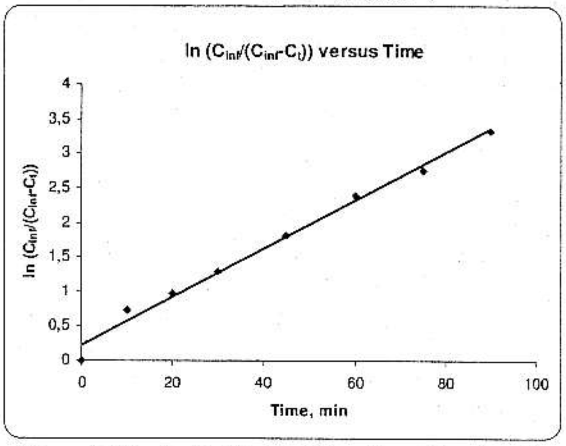

Figure 5: Kinetic Plot for the Extraction of Tongkat Ali

Solid Diffusivity, D_s

The solid diffusion, D_s, can be found from the steady- state model by Spiro and Jago [8] where the observed rate constant, k_{obs}, can be expressed as in equation below. This equation assumes that the diffusion of Tongkat Ali solutes is dominated by solid diffusion as shown work by Sim [2].

$$D_s = \frac{k_{obs}d^2}{2} \qquad (2)$$

where D_s is the diffusivity of Tongkat Ali (m^2s^{-1}), d is the thickness of Tongkat Ali (m) and k_{obs} is the first order rate constant (s^{-1}). This model is based on slab geometry as opposed to the spherical geometry assumed in prior work on Tongkat Ali [2, 7].

Effect of Temperature and Flow rate on Solid Diffusivity, D_s

In the study of the extraction rate, the calculation of the rate constant, k_{obs}, from the experiment data was done through a routine in Microsoft Excel based on the steady state kinetic theory elaborated by Spiro and Jago [8] in Equation 1. The values of rate constant, k_{obs}, as shown in Table 2 were used to calculate the solid diffusivity, D_s using Equation 2. Solid diffusivity, D_s, is directly proportional to the rate constant, k_{obs}, as solid diffusivity, D_s, is the dominant mass transfer factor as shown in work by Sim [2]. Solid diffusivity, D_s, is chosen as primary parameter as it indicates the mass transfer effectiveness. In the calculation of the solid diffusivity, D_s, the particle of Tongkat Ali is considered to have slab geometry. An average of 50 Tongkat Ali particles was measured to determine the thickness of each particle, d, to calculate the solid diffusivity, D_s. The mean value and the standard deviation of the particle thickness, d, were found to be 0.714 ± 0.282 mm.

The effect of temperature and flow rate on the solid diffusivity, D_s, is shown in Figure 6 and Figure 7. Figure 6 indicates that the extraction at higher temperatures increases the value of solid diffusivity, D_s, i.e. it increases the mass transfer rate of solute from the Tongkat Ali particle to bulk solution. Similarly, in plots C of $-\ln k_{obs}$ versus 1/T, the rate constant, k_{obs}, is found to be directly proportional to temperature. Comparable results have been found in caffeine extraction by Jaganyi and Price [9].

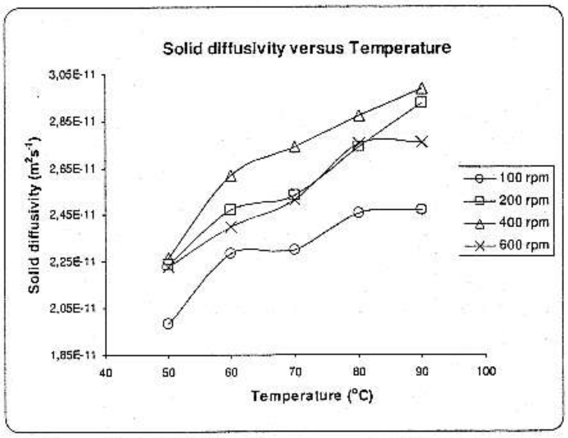

Figure 6: Solid Diffusivity, D_s, against Temperature

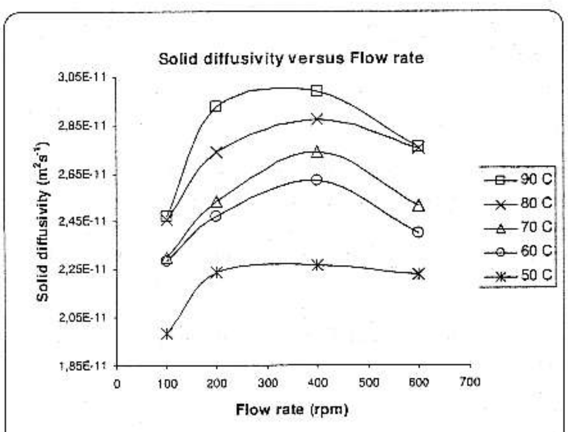

Figure 7: Solid Diffusivity, D_s, against Flow rate

It was also found that the flow rate effects solid diffusivity, D_s, as shown in Figure 7. The results show that the value of solid diffusivity, D_s, is increases in extraction from 100 rpm to 400 rpm but decreases at 600 rpm. It is possible that this is due to the solvent contacting the Tongkat Ali particle more uniformly from 200 rpm to 400 rpm than at 100 rpm and 600 rpm. The exact reason is not clear at the moment.

It can be concluded however that the mass transfer rate of extraction utilizing a recirculating flow extractor is influenced by the flow trend in the extraction vessel. At the optimum flow rate the solvent is able to extract uniformly and at the highest or lowest flow rate, the solvent flow in the extraction vessel may not be uniform.

The range of solid diffusivity, D_s, was found is to be between 1.98×10^{-11} m^2s^{-1} to 2.98×10^{-11} m^2s^{-1} and increases with temperature which is consistent with mass transfer theory. A comparable range of values were also found by Sim [2] between 5.0×10^{-12} to 1.3×10^{-10} m^2s^{-1} for a batch extraction process. Note that the difference of values also stem from the change of solid diffusion calculation based on a slab geometry rather than a spherical geometry in Sims [2] work.

Effect of Temperature and Flow rate on Extraction Yield

In this section, the effect of temperature and flow rate to final yield was analyzed. The percentage yield of the extraction was calculated by equation as shown below.

$$Yield = \frac{Concentration\ (g\ /\ ml) \times (Total\ Water\ Volume - Volume\ Sampled\ \&\ Evaporated)}{Mass\ of\ Tongkat\ Ali\ (g)} \times 100\%$$

The losses in extraction volume were corrected for in the calculations of the final yield. Also, from the extraction plot, it was realized that the equilibrium concentration was not always reached. Thus, extraction time was mathematically extended to 120 minutes through a mathematical approximation of the first order equation based on Equation 1 was used to determine the equilibrium concentration at this time. This final concentration together with the corrected volume was used to calculate the extraction yield.

The result of the experiment shows that the extraction temperature has a direct effect on the final yield as shown in Figure 8. However, there is not a clear relationship between flow rate and final yield for the extractions at 50 to 80 °C. A clear relationship is only observed in extraction 90 °C where the final yield increases consistently with flow rate, but decreases at 600 rpm.

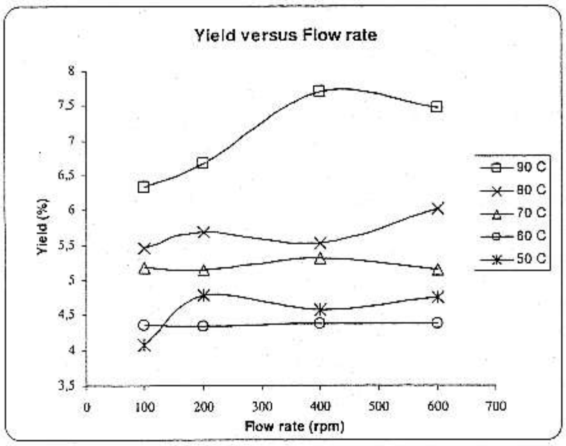

Figure 8: Percentage Extract Yield against Flow rate

The maximum final yield of 7.70 % (w/w) was found at a temperature of 90 °C and a flow rate of 400 rpm. From the solid diffusivity, D_s, calculation in Section 3.5 this is also the point of the greatest solid diffusivity, D_s with a value of 2.98 x 10^{-11} m^2s^{-1}. Therefore, the optimal condition of extraction utilizing a recirculating flow extractor is at 90 °C and 400 rpm (22.47 ml/s). The lowest yield of 4.07% was found at 50 °C and 100 rpm (6.91 ml/s) with a solid diffusivity, D_s of 1.98 x 10^{-11} m^2s^{-1}.

A response surface plot of the effect of temperature and flowrate on yield is shown in Figure 9. Similarly, the plot shows a strong correlation between temperature and yield with yield increasing as temperature increases. Increasing flowrate increases yield but not as strongly as temperature.

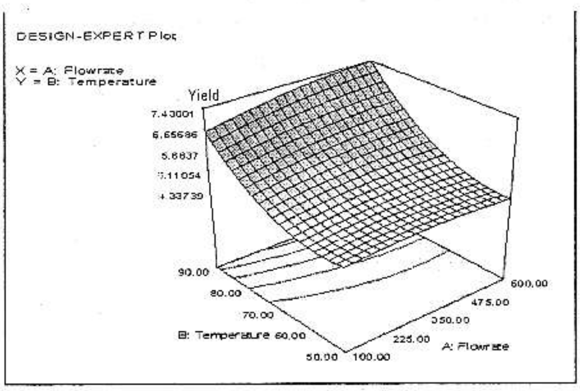

Figure 9: Response Surface Plot

Comparison with Batch Process

A comparison with batch extractor was performed to study the process effectiveness once the optimal extraction condition utilizing a recirculating flow extractor is found. The Batch extraction was performed at 90 °C and utilizes the same particle size (0.5-1) mm and same duration (120 minutes).

Figure 10 shows that the batch extraction can extract more rapidly than recirculating flow extractor and produces a higher final absorbance reading. This is also confirmed through the solid diffusivity, D_s, value for the batch process which is 3.12 x 10^{-11} m^2s^{-1} while it is 2.98 x 10^{-11} m^2s^{-1} for the recirculating flow extractor. However, the rate of water losses through evaporation for batch is higher than the recirculating flow extractor at 1.125 $mlmin^{-1}$ compared to 0.625 $mlmin^{-1}$. Consequently, the extraction utilizing the recirculating flow extractor produces a higher final yield at 7.70 % (w/w) compared to 6.67 % (w/w) for the batch process

Extraction temperature plays an important role that contributes to optimum yield. From results by Kaur [7] and Sim [2] for Tongkat Ali batch extraction, the highest temperature is able to produce maximum yield. For the recirculating flow extractor, the temperature reading is taken in the solvent heating vessel and possibly some heat is lost through the pipe line. Thus, the temperature at the extraction vessel may be lower than the solvent heating vessel. These factors may contribute to the more rapid extraction for the batch process compared to the recirculating flow extractor as the extraction temperature in batch extraction vessel is slightly higher.

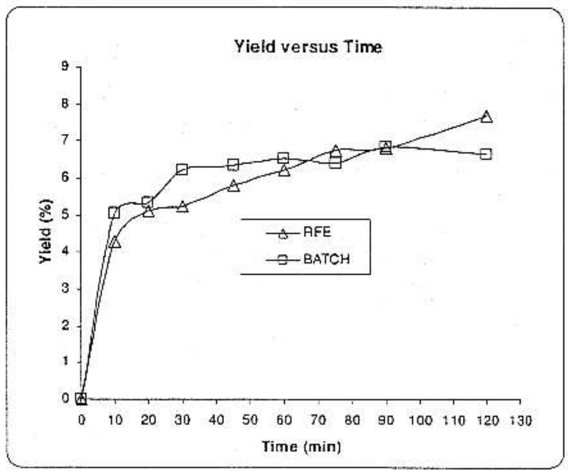

Figure 10: Percentage Extract Yield against Time

Conclusion

From this study, the maximum yield of Tongkat Ali was found to be 7.70 % (w/w) at a temperature of 90 °C and a flow rate of 400 rpm (22.47 ml/s) whilst the minimum yield was found to be 4.07 % at a temperature of 50 °C and a flow rate of 100 rpm (6.91 ml/s). The range of solid diffusivity, D_s, was found to be between 1.98 x 10^{-11} m^2s^{-1} and 2.98 x 10^{-11} m^2s^{-1} and increases with temperature consistent with mass transfer theory. The calculated values were found to be within a comparable range of values found by Sim [2] between 5.0 x 10^{-12} to 1.3 x 10^{-10} m^2s^{-1} for a batch extractor. The optimal point that was selected is at 90 °C and 400 rpm based on the optimal yield and solid diffusivity, D_s.

The comparison with batch extraction at 90 °C showed that the batch extraction was able to extract more rapidly than the recirculating flow extractor. The solid diffusivity, D_s, value for the batch extraction was found to be is 3.12 x 10^{-11} m^2s^{-1} while the recirculating flow extractor had a solid diffusivity, D_s, of 2.98 x 10^{-11} m^2s^{-1} which indicated the difference in extraction rate. However, the extractions utilizing the recirculating flow extractor produce higher final yield than batch extraction namely 7.70 % for recirculating flow extractor and 6.67 % for batch process. This is possibly caused by the higher rates of solvent losses through evaporation for batch extraction.

Acknowledgments

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