Jungbunzlauer

From nature to ingredients®

facts

CITROCOAT® EP – a new effervescent compound for versatile applications

Introduction – Effervescent compounds

Effervescence describes the release of gas from an aqueous solution. The effervescent reaction typically refers to the reaction of an acid with an alkaline carbonate or bicarbonate, more specifically organic acids like citric or tartaric acid and sodium carbonate or sodium bicarbonate, releasing carbon dioxide during the reaction.[1] The endothermic reaction can be observed in various everyday situations, for example during the removal of limestone with acidic cleaners or in diverse food and non-food applications. The effervescence can help with the disintegration of tablets or, as in the case of sweets and beverages, provide a tingling effect.

The first use of an effervescent system in tablet form was in 1957, when the Emerson Drug Company introduced FIZZIES®, a popular drink tablet that fizzed when placed in water. Subsequently the effervescent tablet made its way into a range of pharmaceutical, food and non-food applications.[2] Effervescent tablets combine the convenience of a product in tablet form, to allow for precise and clean dosing, with rapid disintegration during application.

Since the reaction is triggered by the presence of water, the production and storage of effervescent tablets is very demanding. The formation of water during the effervescent reaction promotes a self-sustained process. This means that even minute amounts of humidity can result in problems during manufacture and storage. The fact that the most commonly used acid, citric acid, is hygroscopic exacerbates such issues further.[1] Consequently, the production of effervescent tablets requires a climate-controlled environment. Packaging will need to have low vapour permeability and typically an additional desiccant will be required.[3] As sustainability considerations take on greater significance, plastic-free packaging options are generally gaining popularity. However, these may fail to ensure product stability during storage.

Poor compressibility and segregation tendencies are also common issues in the production of effervescent tablets, while possibilities for a granulation step are limited and give rise to additional costs. Possible solutions include processes that either avoid using water as solvent or can rapidly eliminate moisture to stop the undesired reaction. Direct compression or the separate agglomeration of the acid and (bi)carbonate are other routes often chosen to address these challenges.

With CITROCOAT® EP Jungbunzlauer has developed an effervescent powder that solves many of the issues presented by such products.

CITROCOAT® EP – a new effervescent compound for versatile applications

CITROCOAT® EP consists of coated citric acid (CITROCOAT® N) and sodium bicarbonate. The two reactants are agglomerated using gum arabic as a binder. The agglomeration step improves the powder properties and therefore the performance of the effervescent compound.

The ratio of the effervescent components is formulated to produce a pH of 5.5 after dissolution. The agglomerate size ranges from 200 µm up to 600 µm. Analysis of mechanical stability showed that CITROCOAT® EP maintains its agglomerate structure after moderate vibrational or rotational stress with minimal friability.

The following sections give detailed information on reactivity and storage stability as well as compactibility and processing of the compound.

Higher reactivity and enhanced storage stability of CITROCOAT® EP

The main purpose of an effervescent system is the disintegration of tablets as well as fizzing or foaming in powder formats.[3] Both functions are based on the effervescent reaction.

The reactivity of CITROCOAT® EP was determined by a foaming test. Additionally the carbon dioxide release was measured. For the foaming test 2 g of effervescent compound were applied to 18 mL tap water containing a standardised low surfactant concentration. The resulting foam volume was measured in the cylinder over time. As a benchmark, a dry blend of the raw materials was used (hand mix).

The initial foaming speed within the first 15 seconds was 4.89 mL/s for the hand mix. With the effervescent compound CITROCOAT® EP, reactivity increased by 47% to 7.22 mL/s. This higher reactivity is explained by the powder structure. CITROCOAT® EP is a homogenous agglomerate of citric acid and sodium bicarbonate. Therefore the two reactants are in close contact, leading to a higher reactivity compared to the hand mix when added to water. Additionally, the porous structure provides improved wettability, which is very important in applications where no stirring occurs. Using hydrocolloid gum arabic for the agglomeration process improves foam stability. The foam bubbles resulting when CITROCOAT® EP was used were smaller and more stable over time which effect can be explained by the influence of the hydrocolloid on the foaming system.[4]

While high reactivity is a desirable attribute during the application, this is not the case during storage, where the aim is to prevent the premature reactions of effervescent systems. These would result in a loss of reactivity, significantly influencing performance in the final application, such as slower or incomplete disintegration of tablets. Storage stability was tested at 30°C (86°F) and 50% relative humidity for 14 days. Mass change as an indicator for storage stability was measured daily. In an effervescent powder mixture with uncoated components the effervescent reaction takes place in the presence of moisture. A loss of mass owing to the carbon dioxide release can be observed over time. Using CITROCOAT® N instead of regular citric acid improves the storage stability of effervescent powders (see figure 5). The monosodium citrate coating on the surface of CITROCOAT® N acts as a physical barrier and thereby prevents direct contact of the effervescent reactants. Storage stability is significantly improved. This behaviour can be observed when CITROCOAT® EP and the hand mix as powder or tablet are stored under the same conditions. Importantly, the water-soluble coating of CITROCOAT® N has no impact on the reactivity in the final application. So CITROCOAT® EP combines the benefits of high reactivity when added to water with enhanced storage stability.

Better compactibility of CITROCOAT® EP

Tableting tests were carried out to evaluate the compactibility of CITROCOAT® EP and its performance was compared to a dry blend of all raw materials (hand mix).

In all trials 2% polyethylene glycol (PEG) 4000 was used as lubricant. For the first evaluation step, 5 g tablets with a diameter of 25 mm were compressed with 70 kN compression force. In a second step, the compression force on CITROCOAT® EP was adjusted to achieve a tablet hardness similar to that of the hand mix tablets. Figure 1 shows the results of the tablet hardness analysis.

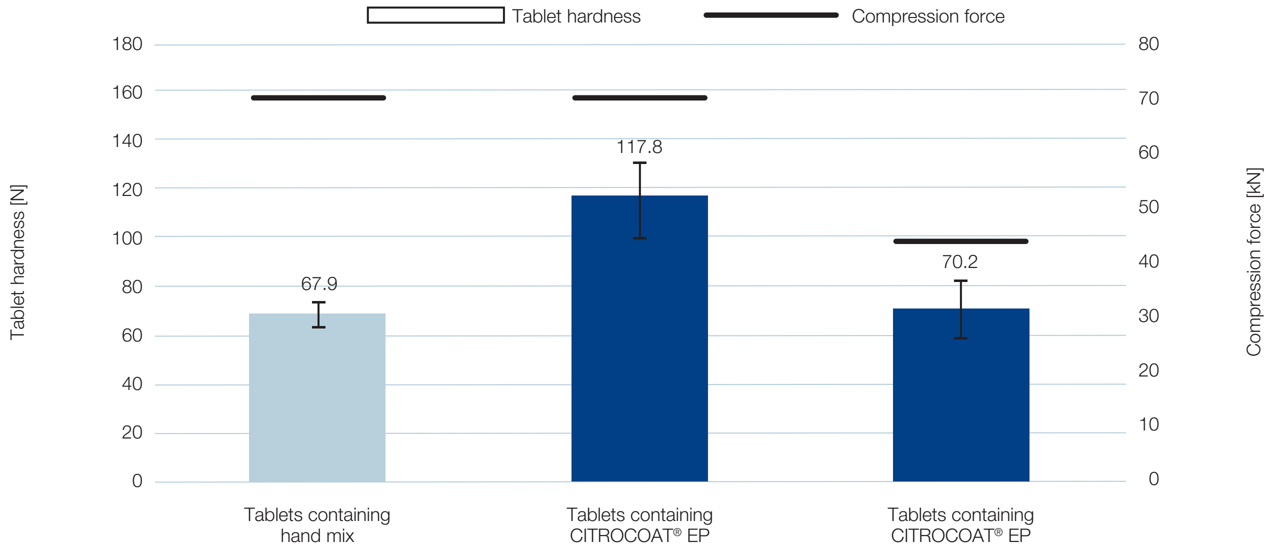

Figure 1: Analysis of tablet hardness with samples containing either effervescent hand mix or CITROCOAT® EP (tablet mass 5 g, n = 20)

Using CITROCOAT® EP resulted in almost double tablet hardness compared to the hand mix. The same tablet hardness for CITROCOAT® EP as for the hand mix was achieved when the compression force was reduced by 37%, from 70 kN to 44 kN. This reduction – through the use of CITROCOAT® EP – leads to increased throughput and efficiency.

Less granular convection with CITROCOAT® EP

Convection and segregation are major problems in transport and manufacturing. These undesired effects are triggered by particle movement in the bulk material. Bigger particles move to the surface whereas smaller particles tend to move downwards.[5]

A segregation test was carried out to evaluate the segregation tendency of CITROCOAT® EP. For this test 2 kg of each blend were placed in a container on a tumbler sieve and stressed for 10 minutes at a speed of 270 rpm. After the segregation test, samples were taken from different locations within the container (top, centre, bottom) and each of the powder samples was compressed with the same compression set-up as described above. The filling depth was kept constant so that the volume was the same for each sample. After compression and analysis of the tablet mass the tablets were dissolved in 200 mL standardised water before measuring the pH of the final solution. Variations in tablet mass and the final pH value are indicators for segregation in the material. The tablet mass of the hand mix and the CITROCOAT® EP from different locations in the container was analysed in relation to the target tablet weight of 5 g (black line). The results are shown in figure 2.

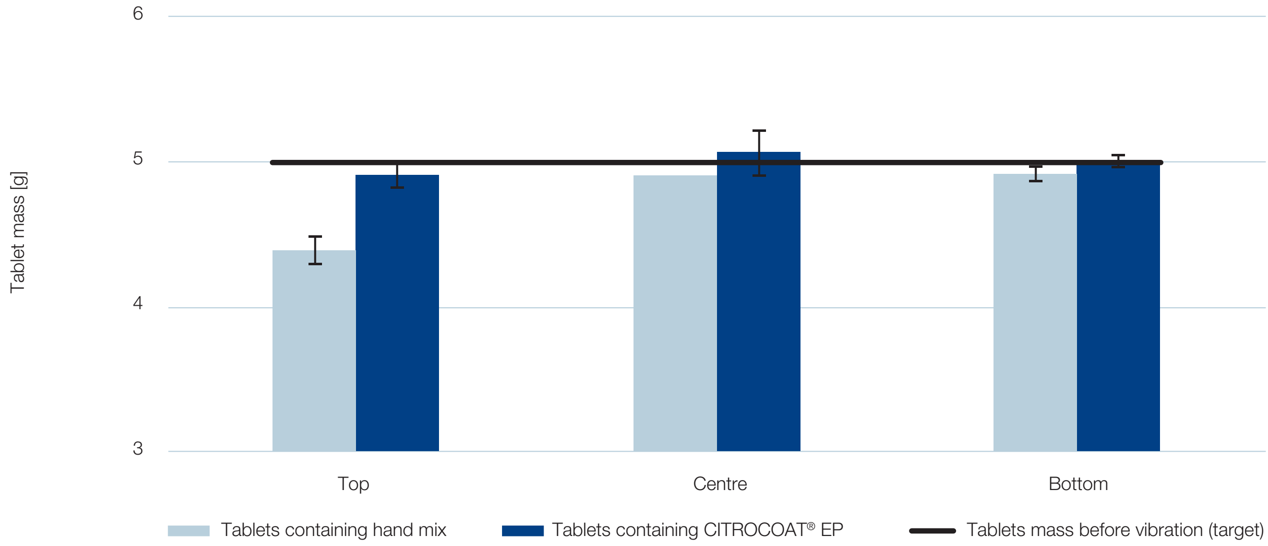

Figure 2: Analysis of tablet mass (in g) from different locations in the container before and after segregation test (n = 20)

Comparison of the tablet mass showed that tablets produced with CITROCOAT® EP from each location had the same weight, whereas the tablets of the hand mix varied significantly. The loss of tablet weight in the sample from the top of the container is an indicator that bigger particles in the hand mix moved to the surface during the segregation test. This led to a lower bulk density of that fraction and consequently to tablets with lower mass.

The pH of the final solution is dependent on the tablet composition. Figure 3 shows the results of an analysis of pH in the hand mix and the CITROCOAT® EP tablets from different locations in the container (in relation to the target pH of 5.5 (black line)).

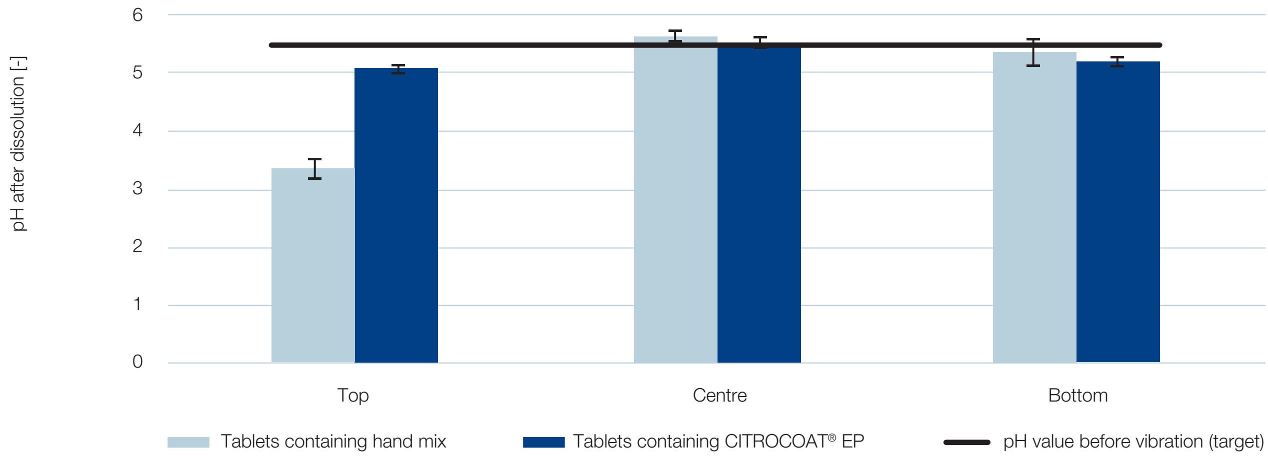

Figure 3: Analysis of pH value before and after segregation test in samples from different locations in the container (n = 8)

For tablets produced with CITROCOAT® EP, the pH showed relatively constant values for all locations. A substantial drop to pH 3.4 could be seen for the hand mix tablets from samples within the top fraction. This indicates a significant change in the composition of the bulk material. These results concur with the results of the tablet mass analysis and are also an indicator that bigger particles, in this case CITROCOAT® N from the non-agglomerated material (hand mix) move to the bulk surface during the segregation test. With an agglomerated effervescent compound, such as CITROCOAT® EP, the segregation potential is significantly reduced, making uniform dosing possible.

To test the performance of the CITROCOAT® EP in a full formulation, tableting tests and analysis were also carried out with multipurpose cleaner tablets.

CITROCOAT® EP in a solid cleaner application

Effervescent systems can be used in diverse applications, for example supplements, cleaning or shower tablets. Currently there is a growing demand and trend towards solid formats – i.e. powder or tablet formats – to significantly reduce a product's volume and weight, potentially meaning less packaging waste and a reduction in the emissions associated with transport.[6]

Furthermore, solid dosage forms offer other benefits apart from sustainability. For instance, the reduction in water activity may limit the need for preservatives – a clear advantage from a clean label standpoint. In addition, novel dosage forms such as tablets create an easier-to-handle product for consumers. This trend towards tablets that can be dissolved at home is also apparent in the home care market.

Some of the most frequently used household cleaners are multipurpose cleaners. They consist of water containing surfactant mixtures, solvents, chelating agents, preservatives and small amounts of perfumes and colorants/dyes.[7] A water-free version of cleaner tablets must include an effervescent system for disintegration. The multipurpose formulation developed at Jungbunzlauer consists of an effervescent system for fast dissolution of the tablets, anionic and non-ionic surfactants for cleaning perfomance and some additional ingredients for special benefits (perfume, colorants, chelating agents). Typical multipurpose cleaner formulations are shown in table 1. The performance of CITROCOAT® EP in the multipurpose cleaner (see formulation 1) was compared to a regular effervescent system of citric acid and sodium bicarbonate (see formulation 2) as well as to a hand mix with the coated citric acid CITROCOAT® N (see formulation 3).

Table 1: Typical multipurpose cleaner tablet formulation

Ingredient	Function	Formulation 1	Formulation 2	Formulation 3
		Weight ratio in tablet	Weight ratio in tablet	Weight ratio in tablet
Sodium Bicarbonate	Effervescent system	6%	30%	30%
Citric Acid (CAA F5020)		-	24%	-
CITROCOAT® N		-	-	24%
CITROCOAT® EP		48%	-	-
Sodium Lauryl Sulfate	Surfactant	22.1%	22.1%	22.1%
C16-18-Fatty Alcohol Ethoxylate	Surfactant	1.9%	1.9%	1.9%
Sodium Benzoate	Preservative	20%	20%	20%
Gluco-delta-Lactone	Chelating agent	2%	2%	2%

All ingredients were mixed and compressed into tablets using a single-punch tablet press. The tablet diameter was 25 mm and the tablet mass 5 g. The tablet hardness of the different formulations was analysed and the results are shown in figure 4.

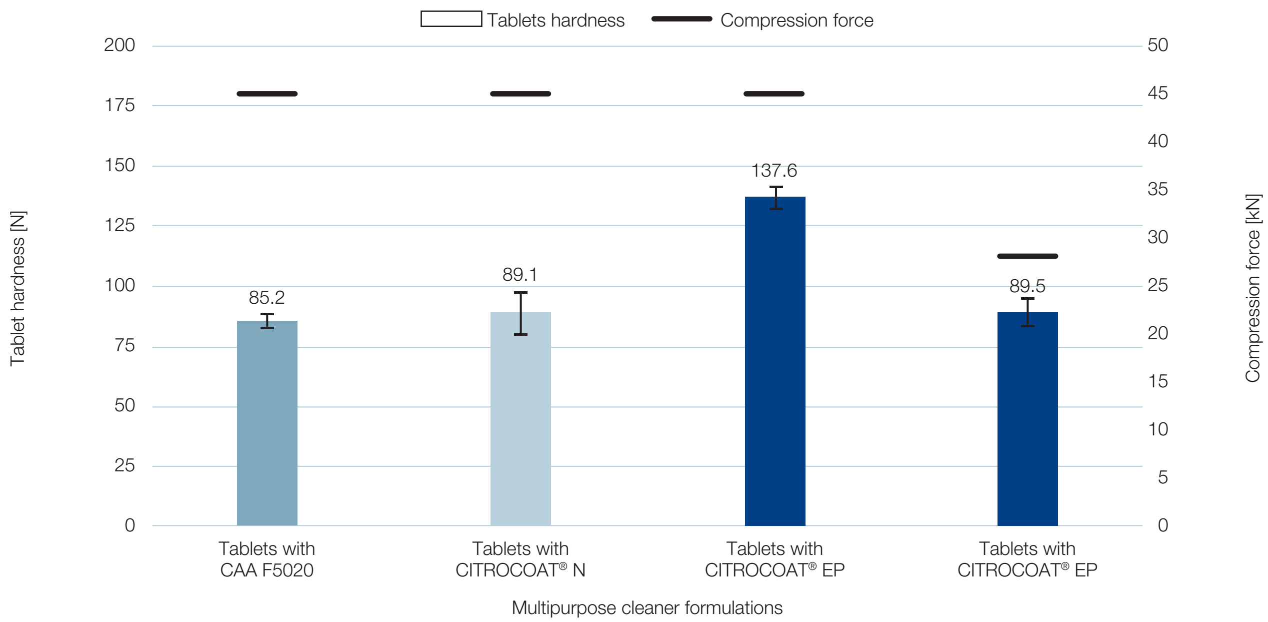

Figure 4: Analysis of the different multipurpose cleaner tablets (comparable to table 1). The bars show the tablet hardness in N (n = 10), while the circles show the compression forces used, in kN

Tableting the formulation with citric acid anhydrous F5020 or CITROCOAT® N using a compression force of 45 kN resulted in a tablet hardness of about 85–90 N. With the same compression force the formulation with CITROCOAT® EP showed an increase in tablet hardness by 55–60% to 138 N. Hence less compression force is needed to produce tablets with the same hardness as the other formulations. In the multipurpose cleaner formulation, CITROCOAT® EP allowed the compression force to be decreased by 60%, to 28 kN.

Another important property of tablets is their storage stability. To investigate this, tablets of all formulations were stored in trays under controlled conditions in a climatic chamber (30°C [86°F] and 50% relative humidity). The mass was measured over time and the change in mass calculated. The results are shown in figure 5.

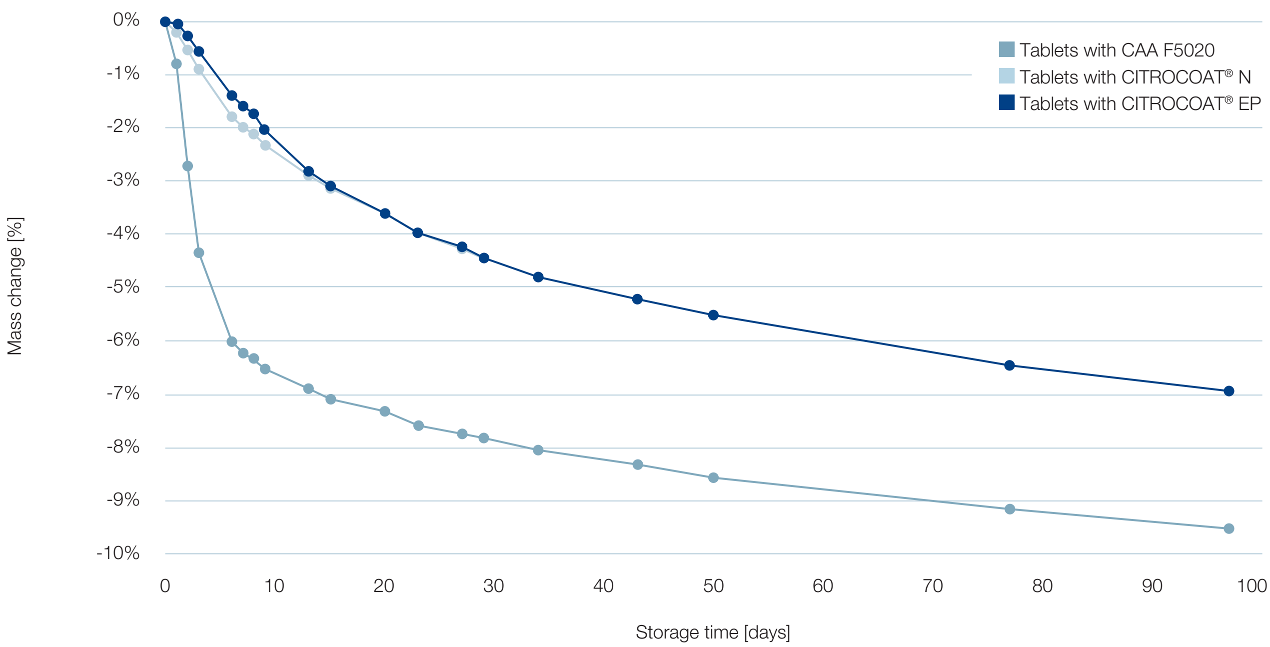

Figure 5: Mass change (in %) over time (in days), tablets (comparable to table 1) stored on open trays at 30°C (86°F) and 50% relative humidity

Due to the effervescent reaction, a mass loss was observed in all formulations. However, while the mass loss for the formulation with regular citric acid was about 10% after 3 months of open storage, the tablets containing CITROCOAT® N and CITROCOAT® EP lost less weight (about 7%). This indicates improved storage stability of tablets with CITROCOAT® N and CITROCOAT® EP.

Summing up, CITROCOAT® EP shows significantly increased compressibility and enhanced storage stability in a multi-ingredient formulation such as a multipurpose cleaner compared to regular citric acid.

Summary

Jungbunzlauer's range of CITROCOAT® products provides a great toolbox for all kinds of applications where citric acid is needed in a more stable form. The new effervescent compound CITROCOAT® EP has demonstrated very good compactibility and improved resistance to segregation, which enables precise dosing and superior processability. High reactivity in contact with water, excellent storage stability and the use of a single component are further relevant benefits. These properties make Jungbunzlauer's CITROCOAT® EP an ideal product for effervescent powder and tablet applications.

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About Jungbunzlauer

Jungbunzlauer is one of the world's leading producers of biodegradable ingredients of natural origin. We enable our customers to manufacture healthier, safer, tastier and more sustainable products. Thanks to continuous investment, state-of-the-art manufacturing processes and comprehensive quality management, we are able to provide outstanding product quality.

Our mission "From nature to ingredients®" commits us to protecting people and their environment.

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