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# Extraction and Characterization of Potassium Carbonate from *Pennisetum Glaucum* (Millet) Straw from Mulai Ward, Borno State Nigeria for Domestic and Industrial Applications

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## Abstract

The investigation of the feasibility of extracting Potassium Carbonate ($K_2CO_3$) from agricultural waste locally is paramount, and can be achieved by sampling and preparing the wastes by ashing, extraction and crystallization, the obtained crystals were analyzed by spectroscopic analysis (XRF, EDX and SEM). The high value of Potassium (K) obtained in both the crystals of millet straw (MS) at sampling areas A and B (MSA and MSB) are 27.3 – 36.66 g/100mg, compared to that of Oxygen (O) 35.4 – 37.7 g/100mg, followed by Carbon (C) 16.6 – 31.7 g/100 mg shadows that the produced crystals met the standard value of the conventional $K_2CO_3$. Conclusively, this research will be useful to those researchers interested in authenticating the fact that the use of $K_2CO_3$ is an important ingredient used in various industrial applications.

## Introduction

Millet is a cereal edit from the Poaceae family, commonly named as the grass family with a seed-like appearance are bunch of small-seeded grasses, broadly developed around the world as cereal crops or grains for grain and human nourishment. Millets are generally annual crops developed and cultivated in India, Northern states of Nigeria, and other Asian and African nations. They run in stature from almost 130 to 300 cm (4 to 8 feet), with the extraordinary case of pearl millet (Pennisetum glaucum), which is brownish in color and thin in thickness has approximately 1.5 to 3 meters (5 to 10 feet) tall and around 2.5 cm (1 inch) thick [31]. They are secured with fiber that is few meters thick.

The inflorescences may be spikes, in which the blooms are on the stalks of approximately breakeven with length along a prolonged pivot. Inside the spikes, the seeds found and eaten as a cereal. With the special case of pearl millet, seeds stay encased in frames after sifting. They are generally ruddy, white and yellow in color, and are

more in fat and tall in carbohydrate. Hulled seeds are ordinarily rich white [30]. Being a portion of the grass family, this lush plant has a long stalk containing less cellulose and is regularly collected for its little palatable seeds. Not at all like most grain crops that like it cooler in temperature, millet develops best in sultrier climate, like rice and corn. Unmistakable combinations of millet are pearl, finger, foxtail, little, and kodo millet which are all developed making the seed as the crucial interest, in spite of the fact that individuals develop millet for distinctive reasons. It is a great cover edit, magnificent nourish for creatures and tamed creatures, scrounge for brushing creatures, and after gather season, blending into your soil for included supplements. A few indeed develop it as a fancy millet since of its great purple foliage [10].

**Taxonomy:**

***Plant Profile***

Kingdom: Plantae, Phylum: Magnoliophyta, Class: Liliopsida, Order: Poales, Family: Poaceae, Genus: *Cenchrus,* Species: *C.americanus*

***Nutritional Composition of Millet***

Millets are staple nourishment for various African countries; be that as it may, they are moo in large scale supplements such as protein and fat but wealthy in vitamins and minerals, gluten-free, fiber, and antioxidant [22]. Millets are extraordinary source of magnesium which diminishes the seriousness of asthma, reoccurrence of migraines, brings down tall level of blood weight and diminishes the hazard of heart assaults. They are moreover a wealthy source of calcium, which ensures bone prosperity, solid compression, blood vessels and fitting nerve work. These supplements play basic parts in human food [18]. The grains are a incredible source of eat less for creating children and eager moms [12]. They are a awesome source of phytochemicals such as polyphenols, tannins, and phytic corrosive which makes a difference bring down the level of cholesterol and decreases cancer chance, tall blood weight, heart illness and diabetes [27]. Other potential wellbeing benefits and therapeutic work of millets are extending in time span of gastric purging and gives roughage to gastro stomach related tract. Millet is in addition known as a soluble shaping nourishment. Soluble based slim down is regularly suggested to get way better ideal wellbeing and anticipate sickness/ maladies [22].

***Mineral Contents***

They have higher sum of minerals such as magnesium, manganese, phosphorus, press, copper and potassium when compared with corn, sorghum and wheat [16]. When comparing millet with other cereals, millet contains 75 % of carbohydrates and is moo in fat (2-5 %) substance than maize, rice, and sorghum [30]. Other potential wellbeing benefits of millets are the headway and repair of body tissue, the anticipation of gallstones, security against breast cancer and assurance against postmenopausal complications and the lessening of chances of childhood cancer [3]. Millets contain 65-75 % of complex carbohydrates, 5.6-12 % protein, fat, 2-5 %, 5-20 % unrefined fiber and 2.5-3.5 % minerals.

***Phytochemical Composition***

Millets have a wealth of antioxidant activity such as phenolic compounds that contains phenolic acids, flavonoids, bioactive and tannins. Bioactive compounds are available in little sums in nourishments, overpowering in entirety grains, natural products, and vegetables and too allow wellbeing preferences in expansion to wholesome esteem [14]. The phenolic compounds of millets are nitty-gritty to have antioxidant, anti-mutagenic, anti-oestrogenic, anti-inflammatory, antiviral affect, and platelet amassing inhibitory movement [1].

The antioxidant exercises of foxtail and proso millets are tall since of their tall add up to carotenoid and tocopherol substance which run from 78 to 366 and 1.3 - 4.0 mg/100 g. They are a extraordinary source of phytochemicals such as polyphenols, tannins, and phytic corrosive which makes a contrast to lower cholesterol and diminishes cancer hazard, tall blood weight, heart illness and diabetes [27]. Other potential wellbeing benefits and helpful work of millets is extending in time span of gastric purging and gives roughage to gastro digestive system. The grain has a incredible dietary regard making it most portion used by routine customers in a tribal community. Its items are rare in the urban ranges as compared to rice ready-to-eat items [18]. The major challenge with millet grains is that the commercial mechanical strategy of handling the grains are not well-known or created as compared to other cereal grains [25].

***Millet Straw***

Millet straws were awesome source of cellulose substance (39.4 %), hemicellulose (23.9 %), and generally moo in lignin (12.8 %), hence speaking to a promising feedstock. The straw coming about from the grain gather is profitable and can be touched straightforwardly by the creatures or utilized in cut-and-carry nourishing system. In Africa, finger millet straw is enormously utilized for making strings and for covering [13].

**Importance**

***Ethanol***

Making ethanol from cellulosic feed-stocks such as grass, wood, and trim residues is a more included prepare than utilizing starch-based crops. Utilization of straw and agrarian squander, such as husk, may offer help to dodge competition between nourishment and non-food utilize of cereals [2].

There are two fundamental pathways to make cellulosic ethanol: biochemical and thermochemical. The biochemical handle joins a pretreatment to discharge hemicellulose sugars taken after by hydrolysis to break down cellulose into sugars. Sugars are developed into ethanol and lignin is recouped and utilized to provide imperativeness to control the handle. The thermochemical change handle incorporates counting warm and chemicals to a biomass feedstock to provide syngas, which is a blend of carbon monoxide and hydrogen. Syngas is mixed with a catalyst and changed into ethanol and other liquid co-products [27].

***Paper***

Millet stalk is an extraordinary rough fabric for mash making due to the reality that straw mash is way better than the hardwood mash. It can be utilized for low-grade items such as cardboard paper, social paper and family paper [29].

***Fertilizer***

The millet straws can be taken care of by refining the fundamental toxins in the papermaking prepare to deliver fulvic corrosive natural fertilizer [11].

***Briquettes***

Millet straw briquettes are great fuel briquettes. This stalk can be blended with Arabic gum to make coal briquettes. Arabic gum is held in a dry mass share of 20 % of the blend. For the arrangement of the gum arable cover, 1 kg of gum Arabic is drenched in 1.5 L of water for one day. After 24 h of inundation, a coagulated arrangement of gum Arabic is produced. The clay is mixed direct with the carbonized biomass powder [17]. A short time later, an amount of water comparable to 40 % of the add up to dry mass (mass of clay and carbonized biomass powder) is poured into the mix until homogenous blend is produced. Briquetting was done physically by a pound in a round and hollow shape of 5.3 cm of breadth. [9].

***Properties of Potassium Carbonate***

| IUPAC Name. | Potassium Trioxocarbonate(IV) |
|---|---|
| Chemical formula | $K_2CO_3$ |
| Density | 2.43 g/cm$^3$ |
| Molecular weight | 138.205 g/mol |
| Melting Point | 891 $^oC$ |
| Boiling Point | Decomposes |
| State | Solid |

***Application of Potassium Carbonate***

Potassium carbonate have numerous employments mainly in fertilizers and cleaning items. It is also used in heat generation [4]. Potassium carbonate is used in food industry in chocolate making, dried fruit production, bakery, powder grade in pharmaceutical industry. It is utilized as a processing agent in cooked products, brewing beer, oriental noodles and additives in drying raisins. It is

also used as a buffer in the production of fruit or wine [11].

***Potassium Carbonate in the Food Industry***

Potassium carbonate is used in various nourishments. It helps to decrease the sodium content in foods, which promotes their health while maintaining a large amount of dough strength [20].

Therefore, this study reports the extraction of potassium carbonate ($K_2CO_3$) from agricultural waste and the crystallization.

## Materials and Methods

### Sample collection and identification

The straws of millet were collected from Mulai Jere LGA of Borno State in two agricultural locations (A and B agricultural areas) on Longitude 13.4333$^o$E 11.7833$^o$N. The dried samples from the two agricultural locations were then sorted and placed in clean polythene bags and transported to the Department of Pure and Applied Chemistry University of Maiduguri, they were then stored at room temperature (28$^o$C) for further analysis**.**

### Calcinations of Agro Waste Sample

The straws were cut (using what machine) into pieces (state the size) and placed into an oven (model and country of make) and heated to a temperature of 400 °C over a period of four hours until properly ashed.

A metal rod with insulated rubber was used to turn the burning straws, to ensure uniform combustion. The ashed samples were homogenized by pounding using a mortar and pestle, and later sieved using ASTM siever [21,6].

### Synthesis of Potassium Carbonate Powder

Two kilograms (2 kg) each of Millet straw location A (MSA), and Millet straw location B (MSB) were soaked in 400 mL distilled cold water for 24 hrs, in further attempt to ensure maximum extraction of the alkali. Subsequently, the slurry were filtered using a filter paper size 2.0 µm to obtain the alkali containing extract.

The extract obtained was subjected to evaporation to dryness using rotary evaporator at 30 $^o$C to 60 $^o$C to obtain a white powder product. The resulting powders were kept in an air-tight plastic bottle, for further analysis by Scanning Electron Microscopy (SEM), X-ray Flourescence (XRF) analyses and Energy Dispersive X-ray Spectroscopy (EDXRS) respectively; in order to examine the purity and morphology of the synthesized potassium carbonate product [26,28].

### Determination of Elemental Composition

The X-ray Fluorescence (XRF) is a method of identifying the crystalline material of a substance for measuring the concentration of atomic elements. It is a non-destructive instrument. Five grams of the sample crystal was filled into a sample holder. The crystal was analyzed using a X-ray Fluorescence (XRF) spectrophotometer (Model, country of make) to detect these elements: Sodium (Na), Potassium (K), Calcium (Ca), Magnesium (Mg), Iron (Fe), Phosphurus (P), Zinc (Zn) and Lead (Pb) [5].

**Determination of Chemical Composition**

Energy Dispersive X-ray Spectroscopy (EDX) a powerful analytical technique used to identify and quantify chemical characterization of a material. The Energy Dispersive X-ray Spectrometer (Model, country of make) was used to determine the chemical composition of the solid. Five grams of the sample was crushed and grinded into a fine powder. The powdered sample crystal was placed in a sample holder and analyzed. These chemicals are: $K_2CO_3$and their oxides.

***Determination of Purity of the Crude Potash***

With the assumption that the potash is mainly potassium carbonate, 3.4 g of the crude potash (crystals) was dissolved in distilled water and made up to markin a 250 mL standard flask. 10 ml was pipetted into a conical flask and then titrated with 0.1 M hydrochloric acid, using methyl orange as an indicator [7]. 5 replicates titre were obtained, from which average titre was calculated.

$2HCl + K_2CO_3 \rightleftharpoons 2KCl + CO_2 + H_2O$

The concentration of of the alkali was calculated from the following expression:

$$\frac{C_AV_A}{C_BV_B} = \frac{2}{1}$$

**Test for Carbonates**

Few drops of dilute HCl was directly added unto the synthesized crystals solutions. HCl reacts with the carbonate releasing carbon dioxide gas(effervescence). Bubbles or fizzing indicates the presence of carbonates adopted by [8].

$K_2CO_3 + 2HCl \rightleftharpoons 2KCl + CO_2 + H_2O$

**Determination of pH**

One gram (1 g) of each crystal sample was weighed into 100 mL of water in a beaker, then stirred for 5 mins, this was allowed to dissolve for 15 mins before the pH of the supernatant solution was determined. The pH was determined using pH meter (Science Demo Model), the solution was monitored and pH was recorded for 5 days and average pH was computed adopted by [8].

**Results and Discussion**

***SEM Analysis***

Scanning electron microscopy was used to obtain particle morphology and size data. This required obtaining specimens with sufficient density to produce a reasonable number of particles in an image at the desired magnifications while minimizing overlap.

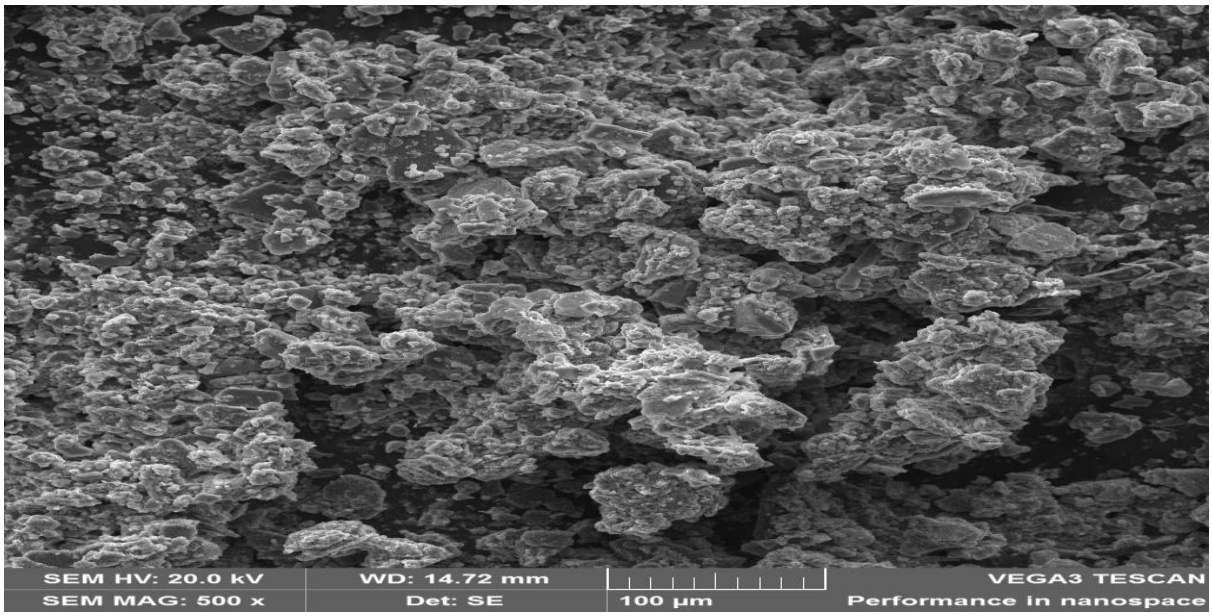


**Figure 1: Surface morphology for sample MSA alkali crystal at *500 magnification.**

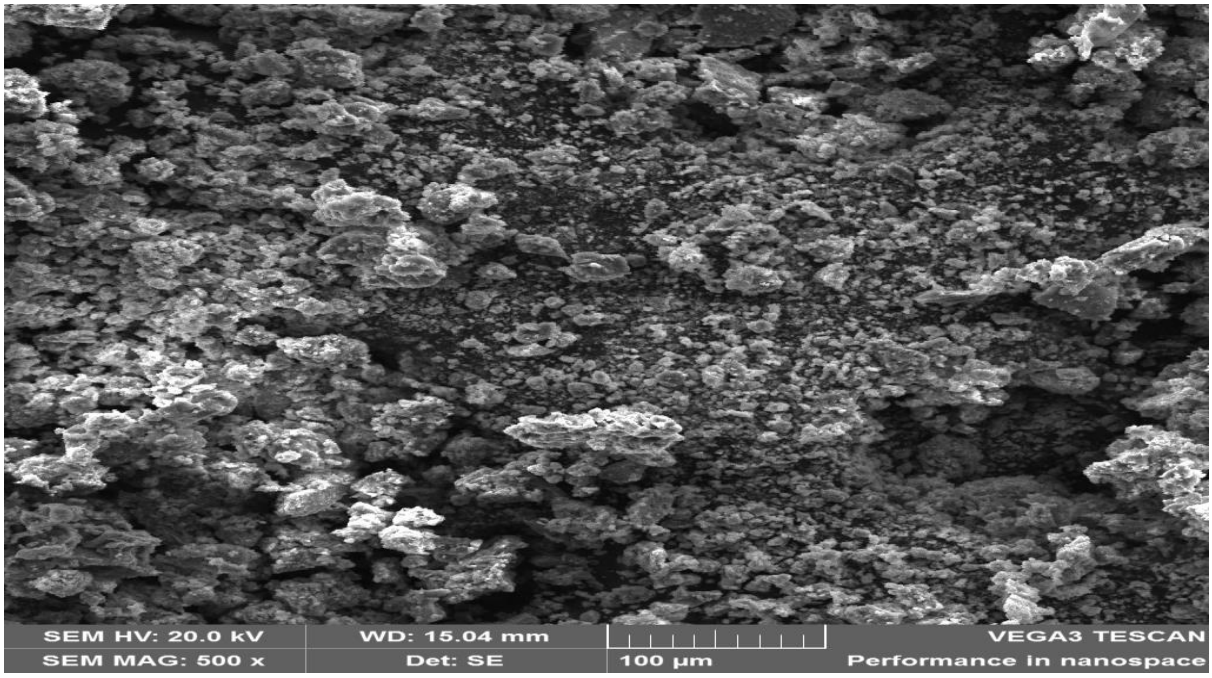


**Figure 2: Surface morphology for sample MSB alkali crystal at *500 magnification**

**Table 1.SEM of the produced crystals**

| **Sample** | Magnification (*) | Feature | Size (µm**)** | Observation |
|---|---|---|---|---|
| MSA | 500 | Particles | 100 | Uniform distribution |
| MSB | 500 | Particles | 100 | Uniform distribution |

SEM images of the produced alkali of MSA and MSB under same magnification using ZEISS sigma model are presented in Fig. 1 and 2. It is obvious from this images that the crystals is orthorombic in nature with an irregular shape having a crystal size ranging from 1-100µm**.** This crystal has a rough surface texture with a porous structure having visible pores which agglomerates of smaller crystals. Crystals that have particle size feature have the ability to be easy flowing reducing its clogging capacity and handling issues.

Crystals with pores have the potential for improved thermal stability. Owing to these features, this pave way for the crystal to be applied in industrial applications like industrial cleaning agents, water treatment, pharmaceuticals, food industry and catalysis as reported by [16]. However, crystals with agglomerated clusters have the capacity to improved flowability, enhanced mixing, better stability, increased density and controlled release. These features make them applied in various industries like pharmaceuticals, food processing and material science as reported by [22].

***Elemental Analysis***

The elemental analysis determines the components of elements present in the samples.

**Table 2: Elemental analysis of the alkali crystals**

| **Sample** | **K** | **Na** | **Mg** | **Al** | **S** | **Si** | **O** | **Cl** | **C** | **P** |
|---|---|---|---|---|---|---|---|---|---|---|
| **MSA** | 36.6 | 0.5 | - | - | 1.9 | 0.9 | 37.7 | 4.8 | 16.6 | 1.4 |
| **MSB** | 27.3 | 0.3 | - | - | 0.6 | 2.1 | 35.4 | 1.2 | 31.7 | 1.3 |

The results of elemental analysis using Thermo Fisher Scientific Niton XL5 model were presented in Table 2. The potassium content was found to be within the range in sample MSA, MSB which is 36.6 and 27.3 g/100 mg. This was evident in the findings of [32]. Oxygen content was found to be the next in value ranging from 35.4 – 37.7 g/100 mg which is close to the expected range of O content (38 – 40 %) as reported by [8]. However, carbon content is next in line with the value of 16.6 – 31.7 g/100 mg which also fall within the expected value of C content (13 – 15 %) reported by [32].. These values of K, O and C that are found close or within the expected range show high purity and $K_2CO_3$ stoichiometric composition. Other elemental contents such as Cl, S, Si and P were found to be impurities in the crystals. These values of elements found in their various concentrations pave way for the crystal to be applied in the industry for the production of fertilizers or plant growth regulators as reported by [32].

According to the concentrations of O and C as stated, it guarantees the crystals industrial application for the manufacture of bio oil, syngas, cement, steel, glass manufacturing, paper bleaching, textile dyeing, soap production, pharmaceuticals and food and beverages as reported by [22]. However, the presence of C and O makes the produced crystals to meet the standard of potassium carbonate that make the crystals to be highly soluble, and to be able to neutralize acids, high melting point and making it highly reactive to form carbon dioxide and water. In other ways, it can be of great importance in the manufacture of glass, soap and detergent, food stabilizer, pharmaceuticals and water softening as reported by [16].

The presence of Cl indicates impurity which implies that the crystals solubility could be altered, pH can also be lowered and this shows the possible effect of environmental exposure. Interestingly, these crystals containing Cl as an impurity can be used in textile industry, water treatment, oil and gas industry, paper production, pharmaceuticals, fertilizer industry and food industry as quoted by [20]. Apparently, the absence of Mg and Al in all the crystals may be due to geological location making this crystals be reduced in reactivity with other substance and exhibit stable pH, despite these, the crystals can be applied in glass manufacturing, soap and detergent industry, pharmaceutical applications and agricultural applications as quoted by [32].

***Oxide Estimation***

Oxide estimation determines the amount of chemicals in form of oxides present in the sample.

**Table 3. The result of oxides presents in the produced alkali crystal.**

| Sample | $K_2O$ | $Na_2O$ | MgO | $Al_2O_3$ | $SiO_2$ | $SO_3$ | $P_4O_6$ |
|---|---|---|---|---|---|---|---|
| **MSA** | 29.62 | 5.66 | - | - | 9.27 | 11.54 | 13.40 |
| **MSB** | 27.54 | 5.44 | - | - | 9.27 | 11.54 | 8.53 |

Oxide estimation using JOEL JED 2300 was presented in **Table 3**. It is obvious that the concentrations of K, Mg and Na oxides were found to fall within the ranges of 27.54. – 29.62 %, and 5.44 – 5.66 %. The presence of $K_2O$ varies widely within the stated ranges, $Na_2O$ content is significant in MSB samples at the ranges of 5.44 %, $Si_2O$ content ranges between 9.27%, $SO_3$ content varies between 11.54 %, $P_4O_6$ is present in both samples between 8.53-13.40 %, MgO, $AL_2O_3$ are absent in both samples this may be due to the geological location of the sampling areas. These ranges in values shows significance in various industries. $K_2O$, $Na_2O$ and $SiO_2$ content suggest suitability for glass and ceramic production as reported by [19].

Likewise, $K_2O$ contents indicates potential use as potassium-based fertilizers as reported by [23]. Similarly, FDA (2012) reported that $P_4O_6$ presence in both samples may indicate suitability for pharmaceutical applications. The absence of MgO and $Al_zO_3$ in both the crystals may be due to geological origin or lack of magnesium-rich minerals in the soil. This may have effect in cement strength and durability, oil and gas reservoir and mineral processing, but can suggests potential catalytic applications as reported by [15].

Despite being absent in the crystals, they can still find their way into various industries like agriculture and animal feed, glass and ceramics, paints and coatings, food packaging, water treatment, chemical and pharmaceuticals, biotechnology, adhesives, energy storage and building and construction materials as reported by [23] that potassium carbonate containing $SO_3$ can be suitable for applications such as wastewater treatment, fuel gas desulphurization, pesticide control, soil remediation and fertilizer production.

## Conclusion

This research work was carried out using standard procedure to test for carbonate, using titrimetric method to determine the purity of the crystals, and also to determine the pH of the crystals. The use of XRF was employed to determine the elemental composition of the produced crystals, EDX was used to determine the estimated oxides of the elements in the

alkali, the morphology of the alkali was determined using SEM at 500 magnification. Furthermore,

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