

Development of a low-cost two-stage technique for production of low-sulphur purified konjac flour

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Abstract: This paper proposes a new kind of low-cost two-stage processing technique aiming at producing low-sulfur purified konjac flour. This technique combines traditional dry and wet processing. This study includes use of advanced techniques in the context of industrial production. The combination of dry and wet processing methods results in the production of purified konjac flour meeting the quality criteria of the top grade as per Chinese industrial standard for konjac flour. The konjac flour obtained by this technique is characterised by a high output and high viscosity on the one hand and low sulphur content and low cost on the other hand.

Keywords: dry and wet processing method, organic and inorganic solvents, anti-browning solution, purified konjac flour, low sulfur

Introduction

China has a long history of using tubers or corms of fresh konjac (*Amorphophallus konjac* K. Koch) and other *Amorphophallus* species for medical treatments and their curative effects were recorded as early as in the late Han dynasty. The fresh corms are bitter and irritating and can be used as medicine against detumescence and anti-toxin to treat sores, snake bites, swollen lymph glands, furuncles (boils) and hernia (Zhang Maoyu, 1989).

The Chinese and Japanese nutrition and public health experts conducted a number of clinical tests of konjac application in human nutrition and came to a common conclusion that the konjac corms which are rich in konjac glucomannan (KGM) are helpful in adjusting the nutritional imbalance (Liu Peiying, 1986; He Jiaqing, 2001). KGM is a hydrophilic dietary fibre. KGM is not digested by humans but has positive effects on human metabolism and health (Sibbel, 2008). The effects of KGM include prevention and treatment of constipation, adjustment of lipid metabolism, improvement of sugar metabolism and immunity regulation. In that respect, KGM is more effective than non-soluble fibres contained in average vegetables (Zhang Chaowu, 1990).

As a result of the above findings, the health

benefits of KGM are being recognised by more and more people. The subsequent expansion of the konjac processing plants and increasing application of this product in the food industry lead to an annual increase of the production of konjac flour by about 10%. Moreover, the current demand for purified konjac flour exceeds the supply. Therefore, the industry attempts to achieve high-output of high-viscosity, low-sulfur content and low cost purified konjac flour (KF).

The main component of KF, KGM, is hydrophilic. In contact with water, it will expand, dissolve, diffuse and become a thick sol. However, KGM will not dissolve in organic solutions such as methanol or ethanol and some inorganic solutions such as NaOH or Na₂B₄O₇. If alkali is added to the expanded sol, a non-reversible, flexible gel is formed.

In order to understand the principles of processing, it is necessary to consider the cellular structure of konjac corms, as shown in Figure 1. Figure 1A shows a succession of cell layers starting from the periphery (top of the picture) towards the centre of the corm (bottom of the picture). Those are: dark brown hardened epidermis, 2-3 subepidermal layers, both without KGM, then the cortex parenchyma, including ordinary cells and large cells (idioblasts) containing KGM (Matsuzaki, 1992). Ordinary cells are small,

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while the idioblasts are oval or round, translucent, large, and their diameter is between 0.25-0.70 mm which is more than 5-10 times that of ordinary cells.

Figure 1B shows a cross section of the corm after staining with I₂ - KI which is a dye indicating the presence of starch: The idioblasts are semi-transparent, without blue coloration, whereas the ordinary cells appear dark blue, which indicates the presence of starch. Hence, during processing of corms into KF, the epidermis, the subepidermal and the ordinary cells must be removed in order to extract KGM contained in the idioblasts.

Table 1 shows the characteristics of idioblasts and compares them with ordinary cells. The processing of konjac corms is based on the differences between these characteristics. For the above reason, the key factor in the production of KF is the separation and extraction of KGM from corm tissue. The traditional processing methods to obtain purified konjac flour are by dry or wet processing which can separate starch granules from larger KGM granules. However, the products obtained by either of the methods have flaws:

The KF produced by the dry method is of low purity, low viscosity and the impurities contained in the powder are not easy to remove. As a result, such product cannot be directly used as a thickening agent and hence is sold at a lower price. As for the wet method, the starch in the cortex cannot be completely separated from KGM and the manufacturing cost is high, which sets some limitations to the application of the KF in fields such as food additives, pharmaceuticals, and fine chemicals.

Features of traditional processing methods producing KF by dry processing method

The process starts by slicing and drying konjac corms. The dried flakes are ground into powder which is called 'common konjac flour'. The latter is then sifted and separated from impurities. This product is called 'common konjac fine flour'. Dry processing method involves two parts: the first is to produce 'common konjac flour' and the second is to grade the 'common konjac flour' into 'common konjac fine flour' and 'common konjac particulate flour'. Since the whole process only involves drying and mechanical separation it is called 'dry processing'. Figure 2 shows the schematic of the dry processing of fresh corms.

The quality of the semi processed materials obtained in the first phase of the dry process (flakes) cannot be controlled since the sliced corms are generally dried either on a sunning floor or on a heated brick bed. The modern processing plants

use vibrating fluidised bed dryers, however, often with direct heating by flue gases. Since different quality of coal is used as a fuel for the fluidised bed dryers, the combustion residues contaminate the dried product. Only the most advanced fluidised bed dryers use heat exchangers. Moreover, in order to prevent discoloration, SO₂ is used, resulting in high sulphur content of the flakes and subsequently of the KF. Besides having high sulphur content, KF produced by the dry process is often characterised by low viscosity (Wu and Zhang, 1994). As a result, most of the product resulting from the traditional dry processing can only be used as third grade flour. Such product often cannot meet the Chinese Professional Standard for Konjac Flour NY/T494-2002 (Ministry of Agriculture P.R.China, 2002), especially with regard to product colour.

In order to obtain good physical and chemical properties of KF, the processor has to prevent it from browning, case hardening and gelatinisation (Huang, 1994). During processing, browning and case hardening would affect the colour of konjac flakes, while gelatinisation would lead to the reduction of viscosity. At present, the traditionally dried flakes often have such defects: The sun dried flakes are often discolored and have low viscosity. The mechanically dried flakes may have acceptable colour but have excessive sulfur content.

Producing purified KF by wet processing method

Wet processing involves using a protective solution for dipping of freshly crushed corms. The protective solution is composed of water, anti-swelling and anti-browning agent. Because KGM is highly hydrophilic, it easily dissolves in water and a sol is formed. Therefore, in the wet process, ethanol is used in different concentrations in the anti-swelling solution in order to control the solubility of KGM in the water-ethanol solution. Because konjac corms contain polyphenoloxidases and tannins, they easily become brown during processing, which lowers the quality of purified flour. In order to prevent it from happening, a variety of anti-browning agents are used that help producing KF of colour meeting the quality standards set out by the industry. In brief, the combined action of the two components of the protective solution protects KGM from expanding in water, turning brown and assures normal operation of equipment. Wet processing involves crushing, grinding, centrifugation and drying. The traditional wet processing technique may be performed in two ways. The first one consists of producing KF directly from fresh corms whereas the second one uses 'common konjac flour' obtained from dry processing

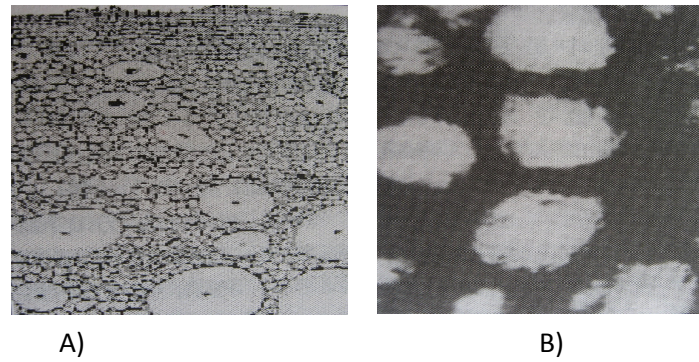


Figure 1. Cross section through a corm of *Amorphophallus konjac* containing KGM.

A) Overall view showing various layers of cells.

B) Staining with I2-KI dye. The large white cells contain KGM whereas the smaller dark cells contain starch.

(Source: Liu Peiyong, 2003)

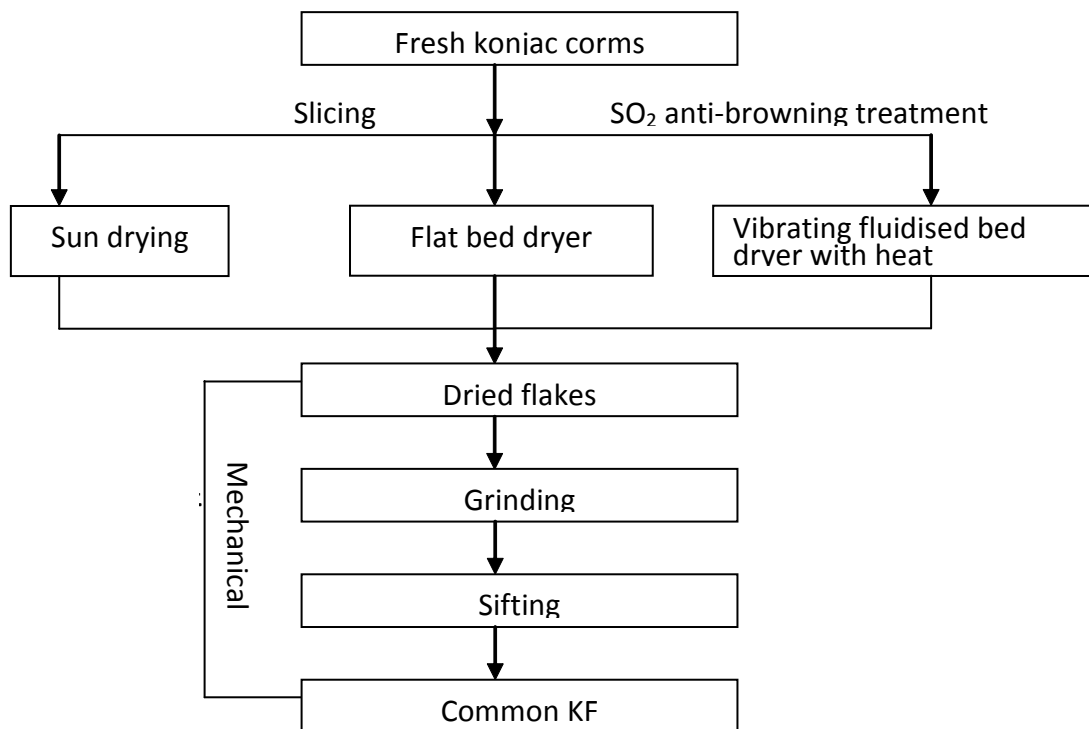


Figure 2. Flow chart showing dry processing of konjac corms

in order to purify it (Zhang, 1994). The cost of the former is high, while the quality of KF obtained by the latter process is not uniform. The protective solution used in the wet process may be of organic or inorganic type. Food grade ethanol is generally used as organic solvent whereas borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) is the most common constituent of the inorganic solution. KF produced using ethanol is of good quality and can be used for various applications such as pharmaceuticals and food, but the cost is high. As for KF produced using borax, it costs less but isn't edible and cannot be used as food.

Given the hygroscopicity of KGM, it has to be dehydrated without delay. Yet, high drying temperature for extended period of time should be avoided as it can significantly reduce the viscosity of KF. The two most commonly used drying methods in wet processing, hot-air and vacuum drying, have their respective advantages and disadvantages. The advantages of hot air drying, generally using a pneumatic dryer, are high speed (a few seconds) and high efficiency. The inlet air temperature in the hot-air method is 120-150 °C. Hence, a short drying time is sufficient and the quality of purified flour is not affected. The disadvantages of the hot-air drying method are the difficulty of recovering the ethanol vapour and the fact that the product has a lingering ethanol smell. In contrast to that, vacuum drying makes easy the recovery and the recycling of ethanol vapour. However, the process speed and the efficiency of vacuum drying are low. The long drying time makes KF darker and its viscosity lower. Therefore, vacuum drying cannot be applied as a single drying stage.

Project aim: devising a process to produce low-sulphur and high-purity KF

Given the shortcomings of the traditional extraction methods, this study aims at improving the production process in order to produce purified KF characterised by high output and high viscosity with low sulphur content and low manufacturing cost.

By combining elements of wet and dry processing in a two-stage technique, as outlined in Figure 3, the total output can be increased whereas the consumption of protective solution and energy consumption can be reduced.

The first step consists in putting crushed corms (but without epidermis or subepidermal tissue) into a series of organic and inorganic solutions at a specific concentration at which the macromolecules can expand without dissolving and becoming mushy. The macromolecules can be broken up gradually and efficiently by a disintegrator and their components

separated. This is the wet processing stage, during which KGM granules will be purified by removing alkaloids, tannins, starch, sulfur and other impurities. Given their high moisture content, the KGM granules have to be dehydrated without delay using a two-stage drying approach combining the advantages of high temperature and those of vacuum drying.

The second step, the dry processing stage, consists of grinding and sifting the KF obtained during the first step following the specifications of the application in which it will be used. A further separation of starch from the surface of KGM granules occurs at this stage.

In brief, the first step is to extract and purify KF by the wet process whereas the second step is to further refine the common KF by the dry process. The objectives of the two-stage technique are:

- to produce KF of a quality similar to that obtained directly by wet processing
- to significantly reduce the production cost

In order to maximise the benefits at an industrial scale, the two-stage technique requires high-speed and high capacity processing equipment for crushing raw corms, grinding and sifting granular material, drying and recycling ethanol (Huang, 2000; Ishii and Kasuga, 1991).

Materials and Methods

Procurement and initial cleaning of konjac corms

The corms of *A. konjac* and *A. bulbifer* were procured directly from the growers in the Dehong and Xishuanbanna regions in Yunnan province (P. R. China). They were harvested and transported directly from the field to the processing plant in order to avoid quality deterioration in storage. Cleaning was done manually in order to remove top buds and roots. Instead of the usual peeling, the authors used a 'non-scraping washing method'. The method is based on the principle that the external cell layers of the corm do not contain KGM but are composed mostly of fibre tissue (Li, 2006). The use of this method helps increasing the recovery of KGM from the crushed corms. Once the corm tissue had been crushed, some of surface fibre tissue was sieved into waste liquid and the rest was dehydrated and ground along with flour granules. During drying, the surface fibres were shrinking and being much lighter than flour granules, could easily be separated from granules in the air classification process.

Sample size

Each experiment was using about 10 kg of corms per batch and was repeated four times.

First stage: wet process

This stage is also called wet milling as all operations leading to the production of purified common flour are dealing with high moisture material.

Pre-treatment in anti-swelling solution

The organic anti-swelling solution was using ethanol. The composition of KGM varies in different species and so does its solubility in the water-ethanol solution. In order to determine the optimum concentration of ethanol for each of the two species, the solubility of KF was investigated in solutions ranging from pure water to 95% ethanol. On the basis of preliminary trials the duration of the solubility experiments was limited to 45 minutes. The inorganic solution was using borax at a concentration of 3%.

Pre-treatment in anti-browning solution

The following four SO₂ releasing agents were used: Na₂SO₃, K₂SO₃, Na₂S₂O₅, NaHSO₃. The concentration range used was between 0.1-0.4%. In order to produce low-sulfur purified flour, vitamin C was also used at a concentration of 6%. The effects of anti-browning treatments were based on the colour index defined in the NY/T494-2002 standard (see Annex).

Crushing and grinding

This process took place in the protective solution and is called wet milling. It involved a grinding mill followed by a colloid mill.

The crushing process was operated in a grinding mill (hammer crusher MFJ-350, 800 kg fresh corms/h, 5.5 kW, 3500 r/min), producing a strong shearing and grinding of the material. The material in the protective solution was forced into a cavity formed by a spinning rotor and fixed stator. A centrifugal force propelled the material to the outside of the rotor, causing intense hydraulic shear that could break agglomerates and homogenise the solids and liquids.

The grinding was carried out in a colloid mill (JM-L50, 100 kg/h, 1.1 kW, 2900 r/min). This process led to further reduction of the particle size of the solids in suspension by applying high levels of hydraulic shear to the process liquid resulting in an increase of stability of the suspension. Due to a thorough action of the grinding and colloid mills, the KF was free of large agglomerated granules as they were in a dispersed state. The process of crushing and grinding of *A. bulbifer* was done in water, as long as it did

not exceed 45 minutes. Beyond this time, the KGM particles started swelling and 30% ethanol needed to be included in the washing liquid.

Washing and dehydration

The supernatant liquid above the ground mass was used as a washing liquid. Washing and dehydration were carried out simultaneously. The dehydrating process took place in a centrifuge fitted with a screen with 149 µm (100 mesh) openings. This operation took about 2 minutes. The washing water was collected in a sedimentation tank with the clear supernatant being recycled as a washing liquid. The liquid with suspended sediment was drained into a waste pool. The water content of the pulp after dehydration was 40% to 60%.

Drying of purified common flour

In order to get the optimum results (complete recycling of ethanol, removal of ethanol smell, high efficiency), a combination of hot-air and vacuum drying has been used. In the first stage, a pneumatic hot air dryer (WGQ30, 1,500 kg wet KF/h, 20 kW) removed the moisture in KF below 12% wet basis. In the second stage, the vacuum dryer (SZG500, 600 kg KF/h, 1.5 kW, 0.03-0.06 Mpa) removed mostly ethanol vapours and smell from the product. The drying parameters for different experiments are summarised in Table 2. The drying parameters of first stage are common for all concentrations of ethanol in the protective solution. In the second stage, the drying time is common for all concentrations of ethanol but the drying air temperature decreases with the increasing concentration of ethanol in the solution.

Recycling of ethanol

In the wet process, significant amounts of ethanol are included in the liquid waste that also includes water, fibre, starch and anti-browning agent. The recycling of ethanol consists of distillation of ethanol from waste liquid. After condensation, ethanol was re-used in the wet process. The ethanol recycler used in the experiments was of continuous type (Jiangsu Changxin Drying Equipment Ltd., T-400, capacity: 120 (kg/h), volume of distillation tower: 1,450 L; height of distillation tower: 7 m, cooling area: 2.2 m², condensation area: 11 m², heat transfer area: 6.5 m²). The conditions in the recycler were as follows: the temperature at the bottom of the tower was 95-105°C, the distillation temperature was 78-88°C, the vapour pressure was 0.08-0.12 Mpa. Under these conditions, the recycling rate of ethanol was at least 97%. The waste liquid left from distillation was drained into waste pool to be used for other applications.

Table 3. Effectiveness of anti-browning treatments in controlling discoloration of KF

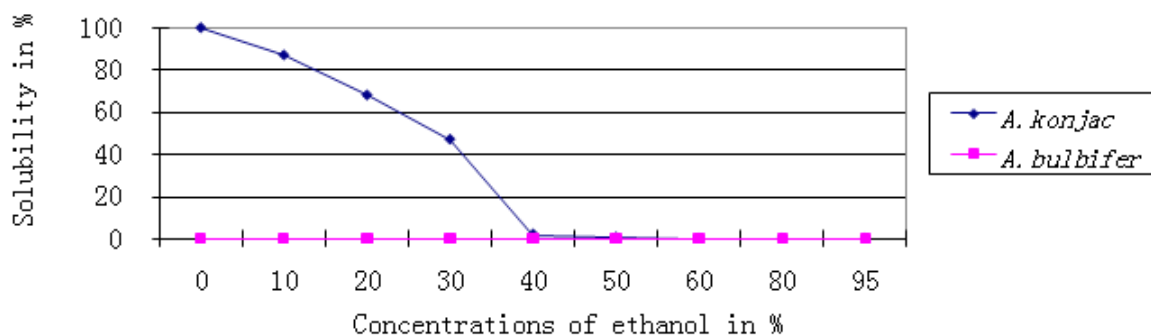
Species	Anti-browning agent													
	Inorganic compounds						Vitamin C							
	Concentration (%)	Na ₂ SO ₃ Colour index*	Residual sulphur (ppm)	Na ₂ SO ₃ Colour index*	Residual sulphur (ppm)	K ₂ SO ₃ Colour index*	Residual sulphur (ppm)	Na ₂ S ₂ O ₅ Colour index*	Residual sulphur (ppm)	NaHSO ₃ Colour index*	Residual sulphur (ppm)	Concentration (%)	Colour index*	Residual sulphur (ppm)
A. konjac	0.1	III	30	III	30	III	30	III	33	III	30			
A. bulbifer		III	30	III	30	III	30	III	33	III	30			
A. konjac	0.15	III	35	III	35	III	35	III	36.5	III	35			
A. bulbifer		III	35	III	35	III	35	III	36.5	III	35			
A. konjac	0.2	II	37.8	II	37.8	II	37.8	II	38.4	II	37.8			
A. bulbifer		II	37.8	II	37.8	II	37.8	II	38.4	II	37.8	6	I	Nil
A. konjac	0.25	I	38	I	38	I	38	I	39.7	I	38			
A. bulbifer		I	38	I	38	I	38	I	39.7	I	38			
A. konjac	0.3	I	42	I	42	I	42	I	44.2	I	42			
A. bulbifer		I	42	I	42	I	42	I	44.2	I	42			
A. konjac	0.4	I	53	I	53	I	53	I	56	I	53			
A. bulbifer		I	53	I	53	I	53	I	56	I	53			

Table 1. Differences between idioblasts and ordinary cells in konjac corm tissue

Attribute	Idioblast	Ordinary cell
Main cell content	Glucomannan	Starch
Hardness of the granules	High	Low, easily break to dust
Granule characteristics	Single granule	Agglomerated granules
Particle diameter (dry)	0.15-0.45 mm	Approx. 0.004 mm
Water-solubility	Easily soluble	Insoluble in cold water

Table 2. Drying treatments

Concentration of ethanol (%)	Hot-air drying			Vacuum drying	
	Inlet air temperature (°C)	Air velocity (m/s)	Drying time (s)	Inlet air temperature (°C)	Drying time (min)
45				130	
55	125	30	2	105	45
65				95	
75				85	

**Figure 4.** Solubility of *A. konjac* and *A. bulbifer* flour in different concentrations of ethanol time≤45min

Second stage: dry process

The second stage, dry milling produced mechanical separation of KGM resulting in the production of purified fine flour.

Grinding and separation of particles

The working principle of dry processing was as follows: konjac flakes are mainly composed of KGM and starch granules. The former account for about 55% and the latter for the remainder of the weight. The two types of granules differ in size and hardness and thus can be separated mechanically. The high-speed grinder (MJ-450, 70 kg/h, with cyclone separator and set of vibrating screens) was first breaking up the softer starch granules and fibre cells (coming from peripheral cell layers of the corms). These granules became disintegrated into tiny, ash-like, particles. During this process, the KGM granules were generally not broken up as they were harder. Due to the difference in size and weight, KGM could be separated from starch particles by cyclonic separation and sifting.

Packaging

According to the accepted industrial practice double-layer packaging was used. The inner layer was a polyethylene film and the outer layer is a woven polyethylene bag. The product was stored in a dry storehouse and was not exposed to sunshine. The temperature was kept below 25°C, and the relative humidity below 65%. The storage time was to be less than 2 years as recommended by the industry.

Moisture content determination

The moisture content of KF was determined according to AOAC (1995).

Viscosity determination

The viscosity of the KF was measured in a 1% solution in water at 30°C using an NDJ-4 viscometer with 4 rotors at 30 rpm.

Data analysis

Statistica 7.1 (Statsoft. Inc. Tulsa, OK, USA) statistical program was used for data analysis. Analysis of variance (ANOVA) was used to determine differences in viscosity between treatments using different concentrations of ethanol. Differences between means were tested for significance by using Duncan's multiple range test with a level of significant of $P < 0.05$.

Results and Discussions

Optimum concentration of ethanol in anti-swelling solution

Figure 4 shows the difference in solubility between *A. konjac* and *A. bulbifer*. It appears that if the wet processing is completed within 45 minutes, the solubility of *A. konjac* (Zhao *et al.*, 2009) depends on the concentration of ethanol in the solution. When the ethanol concentration is less than 40%, the granules of KGM can easily expand and dissolve in the solution, forming a sol. However, once the ethanol concentration exceeds 45%, the granules remain separated. Therefore, the optimum concentration of ethanol is 40-45%. When calculating the amount of ethanol to be added to the solution the moisture content of the fresh corms has to be taken into account. In general, the water content of freshly harvested corms of *A. konjac* is between 75-85% whereas that of *A. bulbifer* is about 70%. However, corms quickly loose water is storage and after ten days the moisture content can drop down to 30% (Ministry of Agriculture P.R.China, 2002). In practice, the weight ratio ethanol: fresh corms is between 1 and 3 (Huang, 2005; Ai and Ai, 2000).

In contrast to *A. konjac*, within this time frame of 45 minutes, the granules of KGM from *A. bulbifer* remain separated and don't dissolve in the solution to form a sol, irrespective of the ethanol concentration. This difference in behaviour of *A. bulbifer* presents the possibility of using water instead of water-ethanol mixture in wet process if its duration is less than 45 minutes.

Effectiveness of anti-browning treatments

The effectiveness of the anti-browning treatments on colour of KF is shown in Table 3. The experimental results show that all four inorganic anti-browning agents gave good protection against discoloration at a concentration of 0.25‰. At this concentration, the sulfurous acid radical remained below $4 \times 10^{-5} \leq 40$ ppm. This complies with the Chinese food standards. The treatment with 6‰ vitamin C resulted in a non-discolored, sulphur-free KF.

Drying of common konjac flour

The effects of the drying treatments on the viscosity of KF are shown in Table 4. The viscometer used was operated at higher rotational speed than that specified in the Industrial standard for Konjac Flour (NY/T494/2002). The higher rotational speed (30 rpm) is currently increasingly adopted by the konjac processing industry. The results show that higher drying temperature reduces viscosity. A higher concentration of ethanol requires a lower drying temperature for the ethanol to evaporate during the

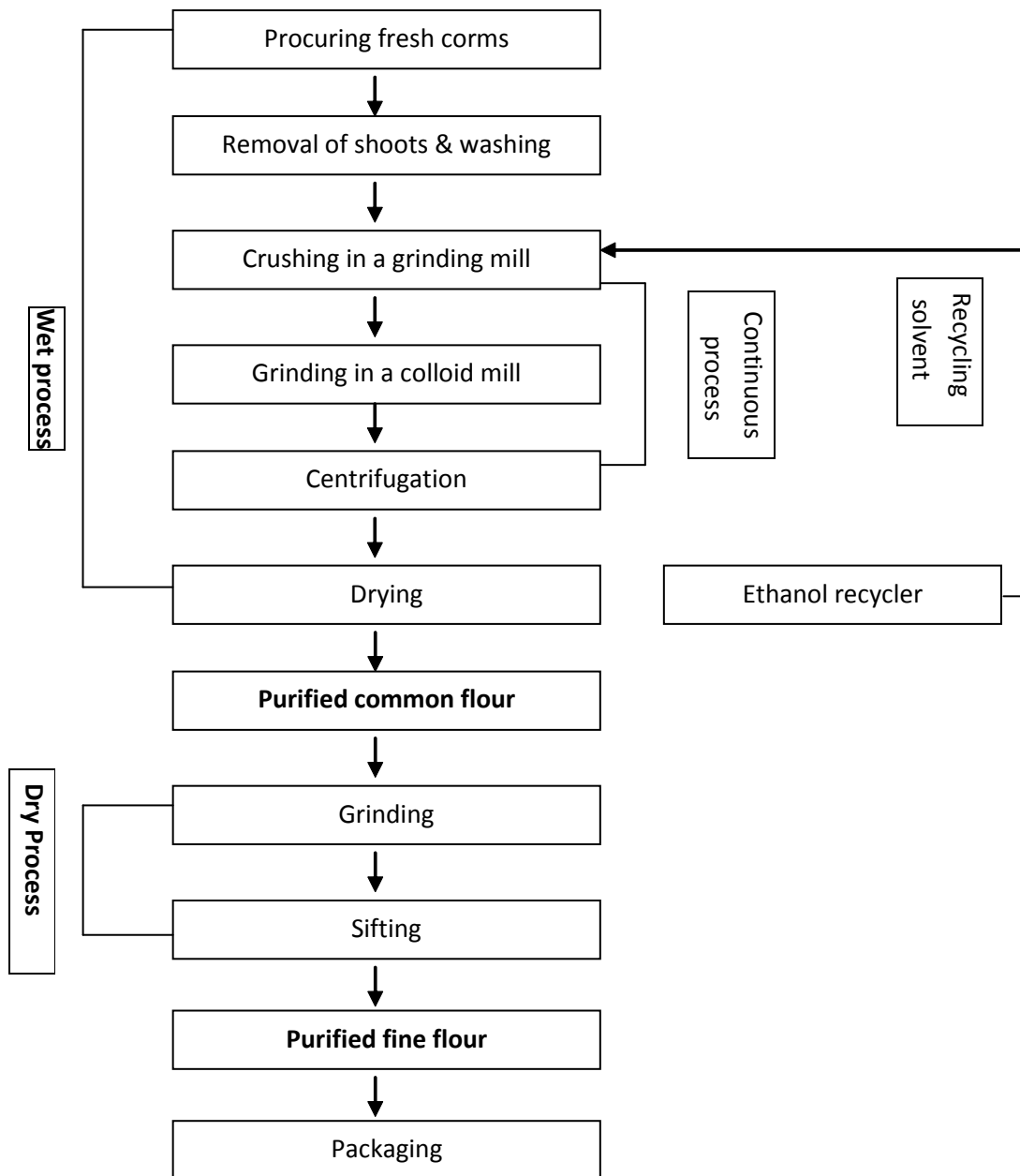


Figure 3. Flow chart of the combined wet and dry process

Table 5. Comparison between traditional and combined wet-dry methods of KF production

Criterion	Traditional techniques			Combined wet-dry technique	
	Dry process	Wet process		Ethanol	Water (<i>A. bulbifer</i>)
		Ethanol	Borax		
Colour	brownish	white	white or yellowish	white	white
Smell	fishy, sulphur-like	slight ethanol smell	subtle fishy smell	slight ethanol smell	subtle fishy smell
Speed of the operation	slow	fast	fast	very fast	very fast
Viscosity (mPa.s) minimum [4 rotors; 30 rpm; 30°C 1% sol.]	8,000*)	22,000	18,000	28,000	28,000
Glucomannan content (%) min.	60	75	70	78	78
Sulphur content (g/kg) max.	2.2	0.4 or sulphur-free	0.4 or sulphur-free	0.4 or sulphur-free	0.4 or sulphur-free
Boron content (g/kg) max.	boron-free	boron-free	1	boron-free	boron-free
Moisture content (% wb) max.	13	11	11	10	10
Ash (%)	6	4.5	5	3	3.5
Sand (%)	0.08	0.04	0.04	0.04	0.04
Compliance with particle size standard (%)	90	90	90	90	90
Production cost	moderate	very high	moderate	low	very low

*) Viscosity of the samples is only an indication of the grade (see standard NY/T494-2002), not an exact value.

same drying time due to its lower specific heat than water and results in a higher viscosity of KF and thus better quality of the dried product. The drying time was the same for all concentrations of ethanol in order to make sure that all ethanol was evaporated. It appears that an ethanol concentration of 65% and a drying air temperature of 95°C resulted in the highest viscosity. However, in spite of high inlet air temperature, the product temperature remains below 70 °C (Li, 2006).

Comparison between traditional methods and combined wet-dry process

The effects of the processing methods on the quality of KF are shown in Table 5. The data in Table 5 show that the combined wet-dry process has a number of advantages over each of the traditional methods used separately. Among the advantages of the combined, wet and dry process, are improved colour and higher viscosity of the product, speed of the operations, and a low production cost.

Conclusions

This paper proposes a new combined wet-dry process to produce purified KF. The materials needed are ethanol, water and anti-browning compounds (inorganic or organic, e.g. vitamin C). By recycling the organic solvent ethanol, the cost of production is kept low and pollution minimised. Moreover, the processing equipment is currently available in konjac processing plants in China. The purified KF produced by the combined wet-dry method is superior to that produced in the traditional way and has potential to gain increased adoption in the industries that use it as a raw material.

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