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**PROCESSING OF STEVIO**

DP/DRK/88/008

DEMOCRATIC PEOPLE'S REPUBLIC OF KOREA

**Technical report: Work performed and recommendations\***

Prepared for the Government of the Democratic People's Republic of Korea  
by the United Nations Industrial Development Organization,  
acting as executing agency for the United Nations Development Programme

Based on the work of Narasimha B. Marla,  
process technologist

Backstopping Officer: T. De Silva  
Chemical Industries Branch

United Nations Industrial Development Organization  
Vienna

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\*This document has not been edited.

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## ABSTRACT

The consultant was briefed by the Backstopping Officer (BSO) in Vienna from 26-28 November 1995. His mission was from 26.11.95 to 8.1.96. He carried out the work as given in his job description (Annex 1).

The process for the extraction of PPSE from stevia leaves had been developed at the Food Research Institute (FRI) with UNDP/UNIDO financial and technical support (1990 & 1992).

The specifications of the pilot plant which were prepared jointly by the NPD and the expert on mission during his earlier mission to the project DP/DRK/88/001 in October/November 1993, were modified to suit the funds available and the pilot plant supplied was generally to these specifications.

The pilot plant of the capacity of 50 kg. per batch of stevia leaves for the extraction of partially purified stevia extract (PPSE) has been installed, tested and successfully commissioned with the active participation of the National Project Director (NPD) and the counterpart staff.

Within the limits of the process, process parameters were worked out including the solubility of PPSE in water at different temperatures, before the commissioning of the pilot plant.

Temperature of extraction was reduced from 65 ° C (at laboratory level) to 55 ° C at pilot level, after initial trial runs in the laboratory.

The percentage of sweeteners in the extract at 55 ° C remained the same as compared to that obtained at 65 ° C. However, the total dissolved solid content in the extract at 65 ° C increased due to the higher solubility of impurities at 65 ° C than at 55 ° C.

Laboratory and pilot plant experiments were based on total sweetener compounds rather than on total dissolved solids content, since this basis is considered erroneous.

Solid to liquid ratio (leaf powder - 15 + 20 mesh : water) of 1:9 was found to be optimum (within the limits of the process developed at FRI).

Washings obtained after washing the cake (twice) were designated as medium micella and light micella respectively. These could be used in a counter-current method.

All the problems in piping connections and the teething troubles associated with the commissioning of the new plant, particular during filtration of extract slurries and foaming during vacuum evaporation of final extract were successfully solved by the consultant.

Concentration of final solution obtained was 1.5 to 2% as against 0.66 - 0.7% in the process developed in the laboratory. No precipitation was noticed during the concentration of final extract 5-40% solution both in the laboratory and at pilot plant.

## I. INTRODUCTION

The climatic conditions in DPR Korea are not conducive for the cultivation of conventional sugar producing crops viz. sugar cane and sugar beet. The country is importing cane sugar, spending a considerable amount of precious foreign exchange.

In the absence of a conventional sweetener viz. sugar in DPR Korea, the plant Stevia Rebaudiana Bertoni holds a great importance to the economy of the country. In recognition of this fact, the Government of DPR Korea has encouraged the Food Research Institute of Light Industry, Academy of Science (FRI) in the early eighties to initiate a research programme to isolate the sweetening compounds from the leaves of this plant and at the same time initiate intense cultivation of the plant.

The naturally occurring stevioside is an intensely sweet disaccharide substance, about 200-300 times as sweet as sugar. It is known to occur to the extent of about 10-20% in the leaves of Stevia Rabaudiana. Extracts of Stevia have been widely utilised in Japan since the mid 1970's as sweetening agents, taste modifiers and sugar substitutes. They are generally accepted as safe food additives.

This plant is extensively cultivated in the DPR Korea and an annual production of 2000-3000 tons of dry leaves has been reported.

FRI with the assistance of UNDP/UNIDO project - DP/DRK/88/008 developed a laboratory scale process for the extraction of partially purified stevia extract (PPSE) from stevia leaves.

The process as reported and worked on bench scale with the assistance of UNIDO experts in 1990 and 1992 is as follows: crushed dried leaves of -15 + 20 mesh, were extracted with water (1:11 solid to liquid ratio) at 65 ° C for one hour and filtered. The total dissolved solids content in the solution was reported to be 4.2% to 4.6%. The stevioside content in the dry matter was about 25%. The filtrate after adjusting to pH to 2.4 with conc HCl was electrolyzed at 45 ° C for 20 minutes using aluminium electrodes at 10V, to precipitate proteins, chlorophyll, dyes etc. which have been extracted from the leaves. The resulting mixture was separated by filtration, the precipitate washed with water and washings added to the main filtrate. To further purify the solution it was passed through a series of ion-exchange columns, first cation, followed by anion and finally mixed bed columns. The columns were washed with water and the washings added to the main solution. The total dry matter in the final solution was reported to be as low as 0.66% to 0.71%. The stevioside content in the dried extract was around 75-80%.

The expert on the current mission with instruction from the Backstopping Officer (BSO) during his earlier mission to the Pyongyang project DP/DRK/88/001, jointly with the NPD and scientists of the stevia research team at FRI, Pyongyang, worked out the specifications of the pilot plant (Annex 2) based on the process developed at FRI. These specifications and the units had to be modified to suit the funds available.

The pilot plant that has been installed and successfully commissioned during his current mission has generally been made to these specifications. The persons contacted by the expert during his mission are given in Annex 3.

Intense process investigations on this pilot plant with stevia leaves is expected to confirm/generate:

- Technical feasibility and economical viability of this process.
- Technology to produce PPSE to reproducible quality and quantity.
- Assess the user industries acceptability of PPSE in their production lines.
- Technical data necessary to scale-up the pilot plant operations to industrial operations in DPR Korea (technology transfer).
- Train engineers and scientists, who could man industrial operations in the future.

- To produce food grade PPSE powder, and also
- To produce standardized food grade concentrated solutions of PPSE.

## II. ACTIVITIES

The expert on mission with the active cooperation of NPD and counterpart staff installed the pilot plant for processing of stevia leaves, according to the layout and the flow diagrams provided by the supplier.

The same was successfully commissioned and the processes of extraction, purification and evaporation to produce partially purified stevia extract from leaves *Stevia Rebaudiana* Bertoni were optimised (Annex 4).

The pilot plant supplied was generally made according to the flow diagram and specifications worked out jointly by the expert on mission (during October/November 1993, to Essential Oil Research Centre, Pyongyang, DPR Korea) with the NPD and the counterpart scientists of the project DP/DRK/88/088 and BSO of UNIDO.

It consisted of the following units:

1. ONE EXTRACTOR : 1.0 M internal diameter, 1.5 M height approximately 1000 litres working capacity provided with anchor type stirrer of 76 rpm fixed.
2. TWO PRESSURE PUMPS : Pumps with motors and starters, one for pumping extracted slurry to the filter press and the other for pumping hot water for washing the cake.
3. TWO CENTRIFUGAL PUMPS : Closed impeller type with motor and starter capacity 1000 l/hr. against total head of 10 M. All contact parts are in AISI 316/PVD5.
4. ONE PLATE AND FRAME FILTER PRESS : Filter Press 450 x 450 mm, washing type, 15 plates and frame chambers made of PVDF and other contact parts in AISI 316.
5. ONE ELECTROLYSER : 1000 x 500 x 500 mm steam jacket (limpet coil) covering the lower half of the tank. 8-A1 electrodes of electrolytic grade 300 x 480 x 5 mm, with electrode suspenders fitted with bolts and wingnuts for the adjustment of the depth of suspension of the electrodes.

Two way electric hoist to remove electrode assembly from tank. Digital indicating thermometer glasswool insulation and covered with 2 mm AISI 304 sheet. All contact parts in AISI 316.

Including AC/DC rectifier, input 3 x 380 V, 60 HZ, output 10V, 250 Amps, switch gear voltmeter, ammeter, etc.

6. ION EXCHANGE COLUMNS : Three ion-exchange columns for cation, anion and mixed-bed exchange respectively:
- Length of each column - 1250 mm.  
 Diameter of cation column - 300 mm.  
 Diameter of anion column - 400 mm.  
 Diameter of mixed bed column - 300 mm.
- In each column a perforated plate provided between two flanges and covered with 8 mesh gauge. Interconnecting pipes and valves with a rotameter to facilitate operation, backwashing and regeneration. Material of construction of all contract parts AISI 316.  
 Resins supplied:  
 Amberlite IR 45 - 400 litres  
 Amberlite 120 - 250 litres
7. 5 NOS. PROCESS TANKS : Process tanks of working capacity 600l each, 900 mm ID. 1100 ht. each with ball valves, glass tube level indicators, loose covers with handles. Material of construction of all contract parts AISI 316.
8. ACID AND ALKALI TANKS : Acid and alkali tanks, capacity 200 l each 600 mm ID, 600 ht. with ball valves, loose cover with handles. Material of construction AISI 316.
9. ONE VACUUM FLASH EVAPORATOR : Vacuum flash evaporator, inclined tube rapid circulation type, suitable for evaporation of heat sensitive organic plant extracts, with a shell and tube condenser, capacity of evaporator 30-50 ltr. of water per hour from extract of 3-4% dry substance to 60% dry substance. The supply includes water ring vacuum pump with motor and starter, vacuum gauge and interconnecting pipes and fittings.
10. SPRAY DRYER : One spray dryer "capacity to match the capacity of flash evaporation" for drying of concentrated extract to water content of 6-8%. Dimensions of drying chamber:
- Diameter - 1000 mm  
 Ht of cylindrical part - 1100 mm  
 Ht of conical part - 960 mm
- Including:
- electrical heat exchanger
  - primary air filter
  - separating cyclone with filter chamber for retaining fine particles.
  - High speed centrifugal atomizer assembly with incremental speed

- adjustment upto 20,000 rpm (maximum).
  - air exhaust fan with motor and starter.
  - control panel.
11. Interconnecting piping and valves. Material of construction of all contact parts of the pilot plant is AISI 316 and gaskets PTFE.
  12. Mild steel support structure.

#### **Some observations regarding the pilot plant supplied**

Checked up the pilot plant unit. The following are some observations and comments:

#### **1. EXTRACTOR:**

The position of thermo-couple provided inside the extractor was much higher than maximum level the slurry could reach. It was therefore, not possible to record the temperature of extraction. A digital indicating thermometer with a thermowell available in the laboratory was temporarily used for the extraction studies.

#### **2. & 3. PUMPS**

As against the order for one pressure pump and three closed impeller centrifugal pumps, the party supplied two pressure pumps and two closed impeller centrifugal pumps.

The change was acceptable, since one of the two pressure pumps could be used for pumping hot water for washing the cake in the filter pressure before its discharge.

Presently, one of the pressure pumps used for pumping the slurry from the extractor is also used for pumping hot water at the end of filtration. The extractor was used for heating water and the same pump was used for washing the cake with hot water. Suitable modifications were made in the piping to let in hot water into the filter press according to the drawing provided by the supplier.

#### **4. PLATE AND FRAME FILTER PRESS**

On test trials with crushed stevia leaves it was noticed that the coarse fibrous particles in the feed, choked the nozzles in the frame corners meant for leading the slurry into the filter chambers and subsequently filled up the entire cylindrical cavity formed by the circular opening provided in all the plates and frames for carrying the slurry into all the chambers of the filter press. This led to the increase in the pressure in the pipe line to 4 bar as shown on the pressure control (PIC) and consequently to the premature shut off of the pump. On opening the filter press the above observations were confirmed.

Another "shoot off" of this problem was that during stoppage of the filtration operation, for cleaning and refitting the filter press, it was noticed that the "elbow" in the pipe line from pump (P1) rising from ground level vertically up, through the "elbow" leading into filter press through pressure control got severely blocked up with solid particles contained in the slurry. This problem was so severe that the elbow had to be cut at one end to clean and rewelded.

This "teething trouble" was solved by resorting to screening the crushed stevia leaves to separate the larger particles (mostly cellulosic stems); only "fines" were used for the extraction. At the beginning of the filtration operation some filtrate collected in the tank (T1), subsequent filtrate was led into tank T2 towards the end of operation, the filtrate in tank (T1) was pumped back into the filter press.



This simple operation gave twin positive results 1) cleaning the pipe line free of slurry, 2) collection of filtrate.

One batch of finely crushed and screened leaf powder after extraction with hot water (55 ° C) and subsequent filtration, caused a serious problem of clogging the filter cloth with very fine particles.

It was solved by replacing the filter cloth with a coarse filter cloth available at FRI and leading back into the extractor keeping the stirrer on, the filtrate for the first 15 minutes containing fine suspended particles. Meanwhile, the hot water, which was made ready in the extractor was pumped into the filter press, for washing the cake. By this action, pressure pump, the piping system leading from the extractor, pump and filter press were thoroughly cleaned. This modified procedure followed in the extraction and filtration operations solved the twin problems.

## 5. ELECTROLYSER

The thermo-couple as in the case of extractor was also placed high. The same procedure was followed by fixing up another thermocouple with a digital indicating thermometer.

In place of a manually operated chain block of 100 kg. capacity, an electrically operated two way (up and down movement) electric hoist has been supplied. This was acceptable. However, the electric hoist supplied was of under capacity. It could not lift the electrode stack by itself. It required additional manual propping.

## 6. ION EXCHANGE COLUMNS AND ION EXCHANGE RESINS

a) Against ordered Amberlite IR 45 (party's ref. no. 940601 dated 27 January 1994). Amberlite IR-A-93 has been supplied. Counterpart scientist at FRI who conducted laboratory scale studies using both resins made the following observations. However, the specifications of the two resins were very similar. It is expected that the performance of both will be comparable on the large scale.

TYPE	IR 45 (OH)	IRA 93 (OH)
Yield of sweetener %	97.3	63.8
Decolouring %	57.9	72.5
pH	6	7

b) Against Amberlite 120 ordered, supply was Amberlite 1200 H.

Though the resins were not acceptable to the NPD and the counterpart scientist, the supplier indicated that the resins supplied are the only ones available at present.

Perforated plates to be fitted on top of each column, have not been supplied.

## 7. 5 NOS. PROCESS TANKS

Generally, they are as per order, however, the UNIDO Purchase Order No.15-4-43/02 was "including glass tube level indicator, complete with stainless steel cocks". None of the five tanks have been fitted with stainless steel cocks.

All glass tube level indicators should be fitted with stainless steel cocks in order to stop loss of solution in case of an accidental damage to the glass tube indicators.

## 8. 2 NOS. PROCESS TANKS OF 200 L CAPACITY

For storing alkali and acid respectively generally as per the order.

## 9. VACUUM FLASH EVAPORATOR

Vacuum flash evaporator and its accessories viz. shell and tube condenser, vapour line, two condensate receivers, watering vacuum pump and interconnecting pipes and fittings for vacuum. The entire evaporator unit was tested and it was noticed that the welded joint at the position just above the valve (46) on the horizontal distillate receiver (C.C.) leaked profusely. This was rectified by proper welding. This could be due to some damage during transport as it was tested before shipping.

Another point to note was that the vacuum evaporator did not have a concentrate receiver. It would have been better to connect a receiver to the source of vacuum, vacuum relief valve and the vacuum gauge even if the unit is designed to be a batch type. The concentrate could then be collected into the receiver without breaking the vacuum and the process continued.

An old 70 l. cylindrical tank located in FRI was being modified to function as a receiver and will be duly connected to the system when completed.

Vide fax dated 17.10.94 from the supplier dated 2/6, item under accessories, one digital indicating vacuum gauge was to be supplied. Actual supply was a dial vacuum gauge.

## 10. SPRAY DRYER

Repeated requests (by fax) beginning 6.12.95 through the office of UNDP to the supplier to send operating instructions of the spray dryer, the control panel and the supply position of metering pump, went unanswered by the suppliers. The suppliers responded only when UNDP Resident Representative telephoned them on 4 January 1996.

The feed arrangement provided to the spray dryer by the supplier was unsatisfactory as per operating instructions for spray dryer 9403-30N page 3/11, 3 operations "material to be dried may gradually be fed to spraying cup. This is done by way of the stainless steel tube, funnel and rubber tube from 5 l. glass flask to be positioned above the dryer. The flow rate is controlled by reducing the aperture of the rubber tube with adjustable steel clamp as known from the laboratory".

Hence, a request was made to get a pumping system.

### Installation

The following connections in the inter-piping were corrected (refer supplier's drawing No.9403-110) with the assistance of FRI counterparts..

- Filtrate from filter press connected to tanks T2m T3, T4 and to tank T1.
- Overflow from electrolyser was connected to tank T4 where as per the above referred drawing it should be connected to Tank T2.
- Outlet from pump P3 was connected only to the electrolyser through valve 50, there was no side connection to the extractor through valve 49.

Another wrong connection that could not be corrected for want of an additional valve was the missing valve (7), connecting filtrate outlet from the filter press to the extractor through valve (7). The pipe line leading the filtrate directly to the extractor was left as such without valve (7). This position may not be a serious handicap in operation of the plant. The valve was requested.

Perforated plates on top of all the ion exchange columns and all the additional items requested after inspection by the BSO and agreed by the supplier have not been received.

### III. CONCLUSIONS

In recognition of the importance of the fact that this plant holds promise to fill in the great gap between the demand and supply of sweeteners in the country and importance of these products, viz. PPSE, stevioside etc. from the plant Stevia Rebaudiana. UNDP/UNIDO stepped in and reinforced the efforts and technical expertise in strengthening the research institute by providing a pilot plant, modern quality control instruments and the services of international experts.

Using the pilot plant of the capacity of 50 kg. per batch (crushed leaf) it was possible to produce partially purified stevia extract and to establish the following:

- It is technically feasible to produce PPSE powder and concentrated solutions, on a pilot plant scale.
- It is possible to reduce the solid to liquid ratio from 1:11 to 1:9; further reduction of this ratio is not considered feasible since the resultant slurry may entail some problems in pumping and filtration.
- It is technically possible to produce higher concentrations of purified stevia extract (before evaporation) from 0.66% to about 1.5 to 2.0%, proportionate reduction in heat energy consumption and increased evaporation capacity, consequently higher yield of PPSE.
- It is feasible and economical to reduce the extraction temperature from 65 °C to 55 °C.

Pilot plant experiments proved that reducing the temperature of extraction resulted in:

1. Considerable reduction in the level of impurities viz. dyes, chlorophyll etc. in the extract.
2. Maintaining the same concentration of total sweetener content.
3. Considerable savings in the heat energy.

FRI is now equipped with basic infrastructure with a moderately equipped R & D laboratory, good quality control facilities, a modern pilot plant and a team of qualified and well trained scientific and engineering personnel who are in a position to produce food grade solutions and powder of a stevia sweetener of standardised concentrations. As such, the Institute has reached a stage to extend continuing technological support to the industry.

### IV. RECOMMENDATIONS

The pilot plant should not only be programmed to produce food grade stevia sweetener powder but also to develop technology to produce related product such as, solutions of stevia sweetener (food grade) of standardized concentration.

Intense studies on the pilot plant should be continued for technology development, to assess its economic viability and to develop parameters for transfer of technology.

Informal discussions with the scientists of the project revealed that they have advanced further in their R and D efforts in the purification and isolation of the following important compounds from PPSE, in the laboratory.

- Stevioside
- Reboudioside A
- Glucosylstevioside

The stevio project which is of socio-economic importance to DPR Korea needs further support both from the Government and UNDP/UNIDO for the logical conclusion to extend the project to produce the above at pilot plant scale to develop industrial scale parameters. The project document (page 6)

noted that "three other products viz. "stevioside", "monoside" and "alphaside" are to be produced. These are not yet commercial products and are produced only on a bench scale, within the stevio research project at FRI, using batchwise processes. Pilot scale equipment to enable the scaling up and improvement of technology for these is not yet available at FRI.

With the setting up of a modern pilot plant at FRI to produce PPSE from stevia leaves completing the first phase, a stage is set to seriously consider launching into the second phase to set-up a pilot plant to produce the above mentioned products as envisaged in the project document. "Following pilot scale studies, a technology package could be formulated by the FRI for improving commercial processing of firstly the PPSE and then stevioside and monoside".

While launching into the second phase augmentation of research laboratory facilities and reinforcement of analytical instruments, setting up a pilot plant to produce the above mentioned products may have to be taken into consideration.

This needs strong support of the Government and UNDP/UNIDO inputs both financial and technological expertise.

The expert on mission recommends the continuation of the project into the second phase to produce steviosides, rebaudioside, etc.

### **Acknowledgements**

The expert acknowledges with thanks, Mr. Tuley De Silva, Special Technical Adviser, Chemical Industries Branch, UNIDO, Vienna, for the advice and generous help extended during briefing on 27 and 28 November 1995, by Mr. G. Faruk Achikzad, Resident Representative, UNDP, Pyongyang, DPRK, for the active interest and advice during the commissioning of the pilot plant at the premises of FRI, Pyongyang on 5 January 1996 and debriefing on 8 January 1996, Mr. Will Scholl, Dpty. Resident Representative, UNDP, for briefing, Mr. Lee Song Ho, Programme Officer, UNDP, for help and assistance, Dr. Kim Dong Su, NPD of the project for actively participating in the erection, testing and commissioning of the pilot plant, Dr. Khim Tae Gyue, Scientist of the project for conducting lab experiments, for help during testing and commissioning of the pilot plant and analytical support, Mr. Khim Jang Sik, counterpart engineer for his assistance during installation and test trials, Mr. Hong Li Suk, M.S. Analytical Chemist and Mr. Khim Yang Choll, Food Processing Engineer, whose assistance in interpretation was of immense help to him.

The expert also wishes to thank Mr. Jong Won Chan, Division Director, GBCIO, for his kind advice and Mr. Jo Jong Nam, Officer GBCIO, for the encouragement given during installation and commissioning.

## UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

DP/DRK/88/008/11-51

## JOB DESCRIPTION

**Post title:** Process Technologist

**Duration:** One month

**Date required:** November 1995

**Duty station:** Pyongyang, Democratic People's Republic of Korea

**Purpose of project:** Enhancement of the efforts of the Foodstuffs Research Institute's Stevio Research Project to develop improved commercially viable processes for the production of sweetening agents from the plant Stevia rebaudiana, based on the laboratory research already carried out.

**Duties:** The expert is required to carry out the following duties in collaboration with the National Project Director and the counterpart staff:

- supervise and direct the assembly and installation of the pilot equipment according to the instructions provided by the supplier;
- commission the different units of the pilot plant;
- test the different stages of the extraction process developed for the pilot production of steviosides;
- conduct trial runs and develop optimal process parameters;
- train the counterpart staff in the use of the pilot equipment and development of process parameters;
- recommend any further improvements for the process and any additional equipment required.

The expert will be required to submit a Final Report embodying his findings and describing the entire work done during the mission and recommendations.

**Qualifications:** A chemical/mechanical engineer with a post-graduate degree and with over 10 years experience in chemical processing at a pilot-scale level. Recent experience with process development of natural products in the context of developing countries will be considered an advantage. The expert will have to possess a displayed ability in productively interacting with national technologists in a developing country.

**Language** English

### Specifications of the Pilot Plant

#### 1. **Extractor:** (Required - one)

Diameter 1.0 m height 1.5 m, 4.25/5 mm thick. Cylindrical dished bottom, flanged open top. Bottom flanged nozzle 50 dia. cross connection. Bottom outlet 50 dia. with flanged ball valve. Anchor type stirrer reaching bottom with 3 HP, 380 V, 60 c/s, 3 phase motor and starter, directly mounted, fixed rpm 60. All contact parts SS316. It should be fitted with thermocouple/sensor and digital indicating thermometer range 20 ° C - 150 ° C. Limpet coil (half tube coil welded to the outer wall of the vessel). Coil to be made of SS304, 3 mm thick to cover bottom dish and 2/3 of cylindrical portion (from the lower end). The vessel to be insulated with 75 mm thick glasswool and covered with 2 mm which SS sheet. M.S. lugs (support brackets).

To be mounted on suitable M.S. structure, platform around with railing and steel ladder and railings. 450 mm clearance from the bottom 50 mm dia discharge ball valve, from the ground level.

Hot water at 65 ° C is the solvent, at atmospheric pressure. Crushed leaves consisting of 80% -15 +20 mesh rest, assorted sizes is the material to be extracted pH of medium -7.

#### 2. **Plate and frame filter press:** (Required - one)

Plate and frame filter press of 450 x 450 mm, 15 chambers, filter cloth, cocks, trough, with suitable pressure pump, motor starter. The unit to be mounted on wheels for mobility. The height should be such that the filtrate to flow by gravity into tanks.

All contact parts SS316.

The filter press should be washing type, to wash the cake with cold/hot water.

#### 3. **Centrifugal pumps:** (Required - five)

Centrifugal closed impeller type with motor and starter 25 dia. suction and delivery. Capacity 1 m<sup>3</sup>/hours against total head of 10 m. Medium: Aqueous extract of pH varying from 1 to 8.

#### 4. **Electrolyser:** (Required - one)

Size 1 m x 0.5 m x 0.5 m, made from SS316, sheet of 5 mm thick. Outlet nozzle flange 25 dia. with 25 dia. ball valve at the lower end on one side, supporting on M.S. legs. 150 clear from the ground level. Jacketed on lower half on all the sides with SS 304 sheet with steam inlet nozzle, pressure gauge, safety valve, condensate outlet, steam trap, steam at 2 kg/cm<sup>2</sup>.

Electrolytic grade aluminium electrodes size 300 x 480 5 mm thick, 80 numbers. Two SS 304 suspenders of the electrodes from the top, with bolts, wingnuts to facilitate adjustment of depth of the electrodes.

Alternate electrodes to be fixed to the suspenders. A pulley block (manually operated) mounted on a rotating vertical shaft (top bent to hold the pulley block 100 kg. capacity and cover the middle of the electrolyser) to lift out the electrode assembly and shift out to one side of electrolyser.

pH of solution 2 (hydrochloric acid is used to adjust pH) temp. 45 ° - 50 ° C.

To be fitted with thermocouple/sensor and digital indicating thermometer. Insulated with 50 mm glass wool and covered with 2 mm thick SS 304 sheet.

**5. AC/DC rectifier: (Required - one)**

Input : 220 V, 60 C/S, 10  
Output: 10 V 250 A

**6. Cation column: (Required - one)**

Generally as shown in the flow sheet  
pH of medium: 1  
Main column 1250 length, 300 dia.

A perforated disc 2 mm dia. holes on 3 PCD covered with 8 mesh wire gauge to be fitted between the two flanges at the outlet end. Perforated disc 2 3 PCD 298 ID over it.

**7. & 8. Anion and mixed bed column: (Required - one each)**

Same as (7) except diameter of the column 400 mm  
pH in anion bed 7-8  
pH in mixed bed 6-7

- a) Anion resin amberite IR-45 0.4 m<sup>3</sup>
- b) Cation resin amberite IR-120 0.25 m<sup>3</sup>

**9. Tanks: (Required - nine)**

Capacity 600 l.

Cylindrical, dished bottom with bottom flanged nozzle 25 dia. fitted with 25 dia. ball valves, open top but fitted with loose covers with handles, with M.S. lugs. All contact parts SS 316. Should be fitted with level indicating assemblies, stainless steel cocks and glass tubings.

**10. Acid tank: (Required - one)**

Capacity 150 ltrs.  
To store dilute hydrochloric acid 7% concentration.

**11. Alkali tank: (Required - one)**

Capacity 200 ltrs.  
To store dilute sodium hydroxide 4% concentration.

**NOTE:**

1. All items namely 4, 7, 8, 9, 10, 11 should be mounted on common mild steel structures. Suitable platforms, ladders with protection railings to be provided for easy access to all the units.
2. All the units namely 1 to 11 should be interconnected with SS 316 quality pipes and fittings generally as shown in the enclosed flow diagram.
3. Flow from cation ion column (7) to the tank (10) and anion column (8) to the tank (10) and mixed bed column (9) to the tank (10) should be connected through rota meters range min 50 l/hr. max. 200 l/hr. With spare glass tubes and 2 rotameter spare.
4. All contact parts in SS 316.
5. All the units should be compactly assembled as a turn-key job.

6. All the internal welds to be ground flush and the vessels to be polished to 180 grit finish. All mild steel surfaces to be painted with two coats of anti corrosive primer and finished with two coats of grey paint.
7. All the gaskets should be of teflon.
8. Two lengths of SS 316 of each size used supply as spares.
9. Six ball valves of 250 size as spares.
10. Additional pipe fittings namely, stainless steel bends, elbows, flanges, bolts and nuts, gaskets to be included as spares.
11. Spare welding electrodes of all sizes used with arc welding as spares.



**LIST OF PERSONS CONTACTED**

Mr. G.F. Achikzad	Resident Representative, UNDP, DPR Korea
Mr. Willi Scholl	Deputy Resident Representative UNDP, DPR Korea
Mr. Lee Song Ho	Programme Officer, UNDP, DPR Korea
Dr. Kim Dong Su	National Project Director, Food Research Institute
Dr. Kim Tae Gyue	Scientist, Food Research Institute
Mr. Jong Won Chan	Div. Director, GBCIO
Mr. Jo Jong Nam	Officer, GBCIO
Mr. Chan Hun Hi	Officer, Department of Foreign Relations, Academy of Sciences
Mr. Kim Young Choll	Food Processing Engineer, Food Research Institute
Mr. Hang Li Suk	M.S. Analytical Chemist, Food Research Institute

TABLE 1

Physical characteristics of stevia leaves and leaf powder

Moisture of dried leaves	...	10% - 13%
Specific gravity	...	1.08 - 1.09 gms/ml
Specific volume (dried loose leaves)	...	30 - 70 kg.
Specific volume (pressed leaves)	...	150 kg.
Specific volume of milled 3-5 mm size	...	55 - 75 kg.
Specific volume of milled 1-3 mm size	...	120 - 140 kg.
Bellow 1 mm size	...	200 - 400 kg.

TABLE 2

Pilot plant extraction

Crushed leaves	...	40 kg.
Water	...	440 litres
Powder to water ratio	...	1 : 11
Temperature	...	55 ° C.
Stirrer	...	76 rpm.
Stevioside in the crushed leaves	...	11.6%

Time minutes	Stevioside (in the extract)	Dry solid %	Yield of stevioside	Purity
45	0.098	2.31		42.4
60	1.03	2.50	85.1	41.2
75	1.11	2.71	91.8	41.2
90	1.13	2.76	93.6	
75 (cake)	0.215	1.51		

Powder to water ratio 1:9

60	1.27	3.10	83.0
75	1.35	3.31	88.0
90	1.36	3.34	88.7

Powder to water ratio 1:8

60	1.37		80.3
75	1.45		85.0
90	1.46		85.3

**TABLE 3 - Pilot Plant****Size composition of powdered leaves and its effect on filtration**

<b>Sl. No.</b>	<b>Size (mm)</b>	<b>Sample (1)</b>	<b>Sample (2)</b>
1.	Below 0.1	3.45	5.97
2.	0.10 - 0.25	19.07	29.16
3.	0.25 - 0.40	27.73	32.87
4.	0.40 - 0.63	27.45	23.54
5.	0.63 - 1.00	16.09	8.46
6.	1.00 - 1.60	4.43	
7.	1.60 - 2.00	0.59	
8.	2.00 - 2.50	0.27	
9.	Above 2.5		

Filter cloth: Polypropylene fibre

Sample (1) cylindrical opening in the plates and frames choked with the fibrous particles.

Sample (2) five particles fouled the cloth. The problem solved by using a coarse filter cloth available in FRI and recycling the filtrate obtained during the first few minutes.

**TABLE 4 - Electrolysis (Pilot Plant)**

<b>Sample mnts.</b>	<b>Time %</b>	<b>Sweetener</b>	<b>Dissolved solids %</b>	<b>Colour OD</b>	<b>pH</b>	<b>Purity of Sweetener</b>	<b>Rate of Decolorization</b>
Extract	-	1.06	2.57	8.63	6.82	41.2	
Electrolyte	10	1.06	2.06	1.03	3.66	51.0	88.1
Electrolyte	20	1.05	1.96	0.600	4.08	53.6	93.0
Electrolyte	25	1.05	1.92	0.544	4.15	54.7	93.7
Cake	-	0.267	16.6				