# Application of Combusion/Pyrolysis for Preparing Ash/Activated Carbon from Fermentation Residue - Distillery Grains as Distillery Effluent Purification Filter

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https://doi.org/10.5109/4797827

出版情報：九州大学大学院農学研究院紀要. 67 (2), pp.197-207, 2022-09. Faculty of Agriculture, Kyushu University
バージョン：
権利関係：

J. Fac. Agr., Kyushu Univ., **67** (2), 197–207 (2022)

# Application of Combustion/Pyrolysis for Preparing Ash/Activated Carbon from Fermentation Residue – Distillery Grains as Distillery Effluent Purification Filter

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(*Received May 8, 2022 and accepted May 10, 2022*)

The study used fermentation waste – sorghum distillery residue (SDR) as a precursor. SDR ash were prepared by combusted directly, and SDR activated carbons (SDRAC) were activated by the method of physics activation with steam. The feasibility of purifying into irrigation water with SDRAC from the effluent after the treatment was investigated. The pH value of alkaline water with different weight percents of SDR ash was about 11.20, and of the SDRAC was between 9.39 and 10.09. The alkaline water impregnated with SDRAC was effective to the alkalizing wine factory sewage and as a role of precipitation process. The suspended solids for the purifying into irrigation water remove about 56 to 77%. The chemical results of chemical oxygen demand, biochemical oxygen demand, nitrate nitrogen, and ammonia nitrogen were removed about 39–90, 58–65, 68.0–97, and 1–21%. The metal and trace elements in water showed that heavy metals, Cr, Pb, Zn, Cd, Ni, Cu, Hg, removed 28–99%, and 31–99%. The results of total bacterial count, coliform group and total organic carbon were about 98.2, 58.6 and 32.4%, respectively. The alkaline water of SDR ash can be replaced the alkalization for sewage precipitation process, and SDRAC is one of the potential filters to purify effluent into irrigation water.

**Key words**: Alkalization, Irrigation Water, Purification, Sorghum Distillery Residue (SDR) Activated Carbon (AC), Wine Factory Sewage

## INTRODUCTION

The Kaoliang liquor is made through a process of steaming the sorghum, mixing it with yeast, fermentation, and distillation. However, a large amount of waste (sorghum distillery residue, SDR) is produced during the distilling process of brewing. At present, the SDR is classified as food processing waste in Taiwan and is mostly treated as an outsourced treatment of industrial waste. In addition to handling a great deal of waste distillery residue, the distilleries also produce abundant waste liquid. After the acid waste liquid is collected through pipelines to the sewage treatment plants, it is preliminarily sieved and rectified, and then neutralized with NaOH solution and mixed with coagulantfor aiding to facilitate precipitation. After the biological treatment and precipitation in the final sedimentation tank, it is directly discharged to the off–island sea. As the Kinmen county (Taiwan, ROC) has insufficient surface storage facilities, the lake, reservoir water, and groundwater are sometimes pumped for agricultural and environmental landscape tree irrigation in low flow season. The distilleries discharge 800–1 000 MT secondary treatment effluent every day. The ideal for this study is the industrial effluent discharged from distilleries that can be purified into agricultural irrigation water, and beneficial to the sustainable water environment ecology. It is the goal of water–saving irrigation because how to recycle the effluent is an important topic.

In general sewage treatment, neutralization and precipitation are common adjustment means of chemical treatment. The neutralizing treatment provides H+ or OH– which adjusts the pH of the solution. The hydrogen–containing acid compounds in water react with alkaline –OH to form water and salt compounds. The alkaline solutions employed in neutralization include NaOH and $CaCO_3$. Afterward, the coagulant aids are used in the precipitation treatment to separate the sewage from the sludge. The discharge is allowed after biotreatment and passing the inspection. The treated water is called "effluent". The pH of charcoals are mostly alkaline when it is in water. In the preparation of charcoals, the carbon single bond is converted into a double bond when the hydrogen atoms are being removed from the precursor surface. This leads to delocalization inside carbon like the effect of Lewis Bases. Hence, the charcoals become alkaline (Zhu *et al*., 2012; Lin *et al*., 2014; 2015a; 2015b). In high–temperature activation, a large amount of alkaline functional groups are formed on the surface of the charcoals and the oxygen–containing functional group are formed due to oxygen transfer. The activation temperature influences the basic functional groups on the surface of the charcoals; consequently, the pH increases after the immersion of charcoals in water (Park and Kim, 2001; Zhu *et al*., 2012; Lin *et al*., 2017).

Activated carbon (AC) is a porous adsorbing material with a nonpolar surface, and has stable chemical properties like resistance to acid, alkali, high temperature, and high pressure, and has a well–developed pore structure (Lin *et al*., 2014; 2015a; Peng and Lin, 2015).

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AC can be extensively used for water purification or adsorbing slight organic pollutants and heavy metals in water, and significantly reduce the physically standard turbidity and chromaticity in water (Anu *et al*., 2006; Lin *et al*., 2015b). AC also can reduce chemically standard chemical oxygen demand (COD) and total organic carbon (TOC) in water (Kunio *et al*., 2001; Seyed *et al*., 2004; Omri *et al*., 2006), and inhibit the biologically standard total bacterial count and coliform group in the water (Ogawa *et al*., 2011; Lin *et al*., 2015b; Lin *et al*., 2017). According to the trade statistics of Customs Administration (CA) of the Ministry of Finance, Taiwan had imported 18 116 MTs of activated carbon (CA, 2014). The research on using AC to adsorb pollutants in aqueous solutions and the research on preparing the agricultural waste with high carbon and high cellulose content into AC by carbonization have been developing for years (Ogawa *et al*., 2011; Lin *et al*., 2015b; 2016). These researches can be used for adsorption of diversified development.

This study tried to use the fermentation waste, SDR, to prepare SDR ash and sorghum distillery residue activated carbons (SDRAC) by direct combusing and different stages of physical activation methods with steam. This was to evaluate the alkalization treatment of chemical precipitation of distillery effluent and the feasibility of using the prepared SDRAC to purify the effluent into irrigation water. The SDR was used as the precursor and prepared into SDR ash and SDRAC by aerobic and anaerobic pyrolysis in different temperature conditions. The yield, and iodine adsorption were determined. The SDR ash and SDRAC prepared from SDR were impregnated to form alkaline water for alkalization treatment of distillery sewage. The pH value, suspended solids (SS), electrical conductivity (EC), and COD were tested to evaluate the alkalization treatment and precipitation effects. The hope is to substitute the chemical agent (NaOH) in the alkalization of sewage precipitation treatment with the SDR ash which was obtained by impregnating SDRAC. The dependence on alkaline agent treatment is reduced, and the dose in the discharge stage can be decreased and the subsequent sewage treatment is assisted effectively.

In the effluent purification stage of sewage treatment plants, the effluent was filtered by the SDRAC from above description. The test properties were divided into physical and chemical properties. The physical properties were water temperature, SS, and dissolved oxygen (DO), oil and grease. The chemical properties were pH value, EC, COD, biochemical oxygen demand (BOD), orthophosphate, nitrate nitrogen ($NO_3$–N), ammonia nitrogen ($NH_3$–N), chloride, total nitrogen, sulfate and sodium sulfate, anionic surfactant, and Na absorptivity and residual sodium carbonate, analysis of metals and trace elements in the water. To evaluate the purification of distillery effluent into irrigation water, the total bacterial count, coliform count, and TOC of the purified effluent were determined. In other words, the effluent was purified by using the adsorption characteristics of SDRAC. The objectives were to purify the treated effluent to the standard of irrigation and reusable irrigation water, and had the significance of sustainable development for the aquatic ecological environment and water resource recycling.

## MATERIALS ANDMETHODS

### Test materials

*Sorghum Distillery Residue*

Sorghum Distillery Residue (SDR) from Kinmen Kaoliang Liquor INC. was washed with deionized water before the preparation of SDR ash and sorghum distillery residue activated carbons (SDRAC) to remove foreign substances, such as sand and soil. The SDR was spread out in a ventilated area, air–dried, and the air–dried moisture content was about 12%.

### Test methods

*SDR ash and SDRAC preparation*

The SDR was combusted in a high–temperature furnace to obtain SDR ash. Absolute–dried weight 40 g of SDR was put in the closed container of the super–high temperature vacuum carbonization and activation equipment (Chimei Electroheat Co., Ltd.). The gas nitrogen ($N_2$; 99.99% purity) was admitted, where the gas flow was 200 mL/min, in order that the container was free of oxygen. The carbonization temperatures were 750, 400, 800, and 850°C and the heating rates was 10°C /min, the carbonization process was completed. In the second stage of activation, deionized water was heated to generate steam, and 90, 120, 200, and 500 mL/h of steam flow rate were used as the steam injection The activation temperatures were 750, 800, and 850°C with 60 and 90 min of the activated duration. In the third stage, it was cooled with 200 mL/min $N_2$ for 4 h to room temperature, and the SDRC yield was calculated (Lin *et al*., 2015a; 2015b).

*Determination of SDR ash properties and SDRAC characteristics*

1. SDRAC yield (%) = (absolute–dried weight of SDRAC/ absolute–dried weight of SDR) × 100
2. Iodine value: Tested according to JIS K 1474 Test methods for activated carbon (Foreign Standard) standard by Japanese Industrial Standard. Iodine adsorption quantity computing equation: $I = (10–K \times f) \times 12.69 \times 5/M$
   Where I: iodine adsorption quantity (mg/g); K: the volume of sodium thiosulfate solution for titration (mL); f: the ratio of 0.1 N sodium thiosulfate solution to 0.1 N iodine solution; M: the absolute–dried weight of SDRAC (0.5 g)
3. SDR ash and SDRAC alkaline water and determination of pH value: The SDR ash and SDRAC specimens in different weight percents (1 g: 200 mL, 5 g: 200 mL, 10 g: 200 mL) were mixed with 200 mL distilled water and impregnated into SDR ash and SDRAC alkaline waters. After 1, 3, 5, and 24 h impregnation, the pH value was determined. The

SDR ash and SDRAC after impregnating SDRAC into alkaline water was used for the effluent purification stage.

*SDR ash and SDRAC alkalization procedure and testing items*

1. Distillery sewage alkalization procedure: In Kinmen Kaoliang liquor INC., the alkalified distillery sewage is rapidly mixed in the rapid mixing tank. The chemical alkaline solution (NaOH), coagulant, and sewage are thoroughly mixed and sent to the slow mixing tank. The velocity gradient makes the particles collide with each other to form larger floc molecules for subsequent precipitating action. This part of the test simulated the treatment mode of sewage treatment plants in Kinmen Kaoliang liquor INC., and used a laboratory magnetic mixing platform to simulate the treatment condition of sewage treatment plants. An amount of 200 mL of sewage was alkalified with SDR ash and SDRAC alkaline water. When the pH value reached the required condition for this test, the sewage treatment was simulated by magnetic stirrers. In other words, rapid mixing at 200 rpm for 5 min (simulating rapid mixing of sewage treatment) and then slow mixing at 60 rpm for 10 min (simulating slow mixing of sewage treatment). Finally, after 10 min precipitation, the result of sedimentation velocity was investigated, and the upper clarified liquor was determined. The simulation was performed with different kinds of alkaline water in the same condition to work out the differences and to evaluate the alkalified distillery sewage.
2. pH value determination: The measuring method for hydrogen ion concentration in water–electrode method (NIEA W424.52A) proclaimed by the Environmental Protection Administration (EPA, 2015) was used for determination. The electrode was corrected by standard buffer solution and wiped up. It was inserted into the distillery sewage, alkaline water, and alkalified sewage specimens, stirred slowly and the pH value was read when the liquid was steady.
3. suspended solids (SS) determination: The detection method (NIEA W210.58A) for total dissolved solids and suspended solids in water, 103–105°C drying proclaimed by the EPA (2015) was used for measurement. Calculating equation: SS (mg/L) = (C – D) × 1000 / V

   Where C: suspended solids and filter weight (g), D: filter weight (g), and V: specimen volume (L)
4. electrical conductivity (EC) determination: The measuring method (NIEA W203.51B) for electrical conductivity in water – the EC meter method proclaimed by the EPA (2015) was used for the test. The EC of distillery sewage, alkaline water, and alkalified sewage samples was determined.
5. chemical oxygen demand (COD) determination: The detection method (NIEA W516.55A) for COD in the water containing high concentration halide ions – potassium dichromate reflux method proclaimed by the EPA (2015) was used for the test. The equation is COD (mg/L) = (A–B) × C × 8000 / V.

   Where A: a blank consumed ammonium ferrous sulfate titrating solution volume (mL); B: water sample consumed ammonium ferrous sulfate titrating solution volume (mL); C: mol concentration of ammonium ferrous sulfate (M); V: water specimen volume (mL)

*SDRAC improved distillery effluent into irrigation water and testing items*

1. Standing method: The SDRAC (10 g) and distillery effluent (100 mL) were mixed in the weight percent of 1: 10 (w/w%) and left for 120 min (Ogawa *et al*., 2011; Lin *et al*., 2015a; 2015b). The water specimen [specimen code: prepared condition–testing item (stand)] treated by SDRAC was tested according to the irrigation water quality standard.
2. Filtration method: The SDRAC was put in the glass funnel. The effluent flowed from the top to the bottom under gravity. The flow velocity was controlled by a valve. The weight percent of the SDRAC (10 g) to distillery effluent (100 mL) was 1: 10 (w/w%) and the flow velocity was 5 mL/min (Ogawa *et al*., 2011; Lin *et al*., 2015a; 2015b). The treated water specimen (prepared condition–filter) was evaluated against the irrigation water quality standard.
3. Irrigation water quality testing items
   (1) Physical properties items included water temperature: the water temperature testing method (NIEA W217.51A) proclaimed by the EPA (2015). SS: The detection method (NIEA W210.58A) was same the above description. Dissolved oxygen (DO): The detection method (NIEA W455.52C) for dissolved oxygen in water – electrode method was used. Oil and grease: The detection method for grease in water – Soxhlet extraction gravimetric method (NIEA W505.51C) was used. The tests of SS, DO, and grease were performed as per the guidelines of the EPA (2015).
   (2) Chemical properties items included: The measuring methods for pH value (NIEA W424.52A), EC (NIEA W203.51B), and COD (NIEA W516.55A) were same the above description. Biochemical oxygen demand (BOD): the detection method (NIEA W510.55A) for biochemical oxygen demand in water was used. Orthophosphate: the detection method (NIEA W463.50B) by using the discrete analytical system colorimetry. Nitrate nitrogen ($NO_3$–N): The detection method (NIEA W419.51A) for $NO_3$–N in water – spectrophotometer method was used. Ammonia nitrogen ($NH_3$–N): The detection (NIEA W448.51B) of $NH_3$–N in water – indigotic colorimetry was used. Chloride: the detection is by the spectrophotometer method (NIEA W408.51A). Total nitrogen: the water–

quality determination of total nitrogen – alkaline potassium persulfate digestion UV spectrophotometric method was used. Sulfate: the detection method (NIEA W430.51C) for sulfate in water – turbidity method is used for measurement. Anionic surfactant: the detection method (NIEA W525.52A) for anionic surfactant (methylene blue active substance) in water – methylene blue colorimetry was used. All tests were performed as per the guidelines of the EPA (2015).

(3) Determination of sodium absorptivity ratio (SAR) and residual sodium carbonate (RSC): according to the detection method (NIEA W449.00B) for alkalinity in water – titration proclaimed by the EPA (2015).

(4) Determination of metals and trace elements in water – metals and trace elements in water: Tested by Inductively coupled plasma–mass spectrometer (ICP–MS).

*Biological tests of irrigation water quality items*

1. Total bacterial count: The detection method (NIEA E204.55B) for the total bacterial count in water – mixed dilution method proclaimed by the EPA (2015) was used.
2. Coliform group: The coliform group contains galactosidase and glucuronidase simultaneously, and the colony can be red and dark blue (or purple). The other enteric bacteria without these special enzymes are white or colorless colonies in the culture medium (Lin *et al.*, 2015b). Equation is: coliform count (CFU/100 mL) = [(red colony + violet colony) × 100] / water specimen volume × dilution factor
3. Total organic carbon (TOC): The detection method (NIEA W532.52C) for TOC in water – peroxy–pyrosulfate thermal oxidation/infrared determination proclaimed by the EPA (2015) was used.

*Statistical analysis*

The result was represented by the average value (standard deviation). Statistical Product and Service Solutions (SPSS) 12 was used for statistical analysis with Duncan's multiple range analysis, various treatments were represented by ($\rho$<0.05), and different English letters represent significant difference.

## RESULTS AND DISCUSSION

### SDRAC characteristics

*Yield*

Table 1 shows the yield of SDRAC prepared from SDR in steam–activated conditions. The yield of activation temperatures of 750–850°C was 19.11–28.91%. The yield of SDRAC decreased as the activation temperature increased. This is because the volatile organic compounds in the precursor, tar dissipation, and gasification of carbon are increased (Teng and Hus, 1999). The yield decreased when the temperature was 800°C. This is due to the fact that the activation is effective and the tar can be removed effectively (Lin *et al.*, 2015a and 2016). In addition, the yield decreased as the steam flow rate increased at temperatures higher than 800°C. The reason is the steam activation collapses carbon or/and the carbon surface is oxidized (Aworn *et al.*, 2008; Lin *et al.*, 2014; 2015a; Peng and Lin, 2015).

*Iodine value*

The pore dimension of activated carbon can influence the adsorption of substances. The iodine value can be the non–polar molecule adsorption index of activated carbon. The diameter of the iodine molecule is 0.56 nm. It can be the value for judging activated carbon micropores and larger micropores (Teng and Hus, 1999). According to the iodine value results, the SDRAC prepared by using the same steam flow rate at activation temperatures of 750, 800, and 850°C were 377, 547, and 680 mg/g, respectively. It is observed that the iodine value of the SDRAC increases with activation temperature. The steam flow rate is a factor that influences pore formation (Lin *et al.*, 2015a and 2015b). As the injected gas quantity and activation temperature are increased, the micropores of the prepared activated carbon are widened continuously adjacent to the micropore wall and collapse into larger micropores or mesopores due to the loss of ignition loss (burn out). The pore dimension or volume is increased (Aworn *et al.*, 2008).

### Analysis of alkalization of SDR ash and SDRAC

*pH value change*

To investigate the optimal impregnation time, this study used the impregnation condition of 10 g: 200 mL for multiple repeated evaluations. The relationship between impregnation duration and pH value is shown in Table 2. The T850–90–500 and SDR ash had the highest pH values after 1 h of impregnation which were 10.09

**Table 1.** Yield and iodine value of SDRAC with different preparation conditions

| Specimen [1)] | Yield (%) | Iodine value (mg/g) |
|---|---|---|
| T750–60– 90 | 27.99 (0.01) [gh 2)] | 261.67 (0.00) [a] |
| T750–60–120 | 27.36 (0.03) [fg] | 364.92 (10.15) [b] |
| T750–60–200 | 28.91 (0.04) [h] | 377.02 (5.86) [b] |
| T750–90–120 | 27.00 (0.01) [g] | 464.96 (5.86) [c] |
| T800–60–200 | 24.73 (0.00) [e] | 547.70 (10.15) [d] |
| T800–90–200 | 23.28 (0.01) [d] | 614.03 (5.86) [e] |
| T850–60– 90 | 25.30 (0.01) [ef] | 406.76 (5.86) [b] |
| T850–60–120 | 24.92 (0.01) [e] | 497.96 (0.00) [c] |
| T850–60–200 | 22.43 (0.01) [cd] | 622.32 (0.00) [e] |
| T850–60–500 | 20.93 (0.01) [b] | 672.57 (0.00) [e] |
| T850–90–120 | 21.07 (0.01) [bc] | 640.84 (0.00) [e] |
| T850–90–200 | 21.03 (0.01) [b] | 680.35 (5.86) [f] |
| T850–90–500 | 19.11 (0.01) [a] | 774.09 (5.86) [f] |

[1)] Activated temperature (°C) – Activated duration (min) – steam flow rate (mL/min)

[2)] Mean (standard deviation) by Duncan's multiple range analysis at 5% significant level, and different English letters represent significant difference and 11.23, respectively. The SDRAC had the highest pH value after 1 h impregnation. The SDRAC powder had the highest pH value after 3 h. The T850–60–200 (powder) had the highest pH of 9.68. For a subsequent test, the alkaline water after 1h impregnation of T850–90–500 and SDR ash was used as the specimen for the alkalization treatment. The T850–60–200 (powder) was selected and directly put in the sewage as a control group of alkalization treatment.

*Basic properties of distillery sewage*

Chemical precipitation is an important mean to treat sewage and the water contaminants can be reduced effectively. To know the chemical precipitation treatment process of distillery sewage, the sewage specimen were taken from three parts in Kinmen Kaoliang liquor INC., and their properties were determined. Table 3 shows the pH value, SS, EC, and COD. The sewage precipitation treatment used NaOH as a chemical alkaline solution to increase the pH value to 5.40. With the assistance of coagulant and coagulant aids in the rapid mixing tank, the suspended pollutants were dissolved out of the sewage. When the SS increased from 1,963.33 mg/L to 2,573.33 mg/L, the floated suspended solids were scraped with gas by pressurized flotation. When the SS decreased to 162.33 mg/L, the EC and COD increasd from 0.82 mS/cm and 98.02 mS/cm to 2.58 mS/cm and 100.43 mS/cm, respectively. Therefore, the EC and COD values increased after the NaOH, coagulant, and coagulant aids were applied to sewage treatment.

*Analysis of sewage after simulation test*

The treatment model of sewage treatment plants was simulated. Four different alkalization methods were used to alkalify the sewage. It was compared with the sewage specimen after precipitation in the pressurized flotation tank for distillery sewage treatment, as shown in Table 4. The pH value is closely related to coagulative precipitation (Adin *et al*., 1998). The pH value not only influences the efficacy of the coagulant but also reduces the repulsion between suspended particles. The suspended particles are likely to agglomerate and precipitate effectively (Benefield *et al*., 1982). The four alkalization methods were able to neutralize sewage precipitation treatment. The pH value of sewage increased from 3.14 to 5.11–5.36 and there was no significant difference from the pH 5.40 of the sewage specimen from the flotation tank.

After the precipitation in simulation test, the SS of SDRAC–Water decreased from 933.33 to 277.33 mg/L which was reduced by 656 mg/L. The SS of SDRAC powder decreased from 933.33 to 583.33 mg/L which was a reduction of 350 mg/L. There was no significant difference between the SS of the other alkalified sewage specimen and the sewage specimen from adjustment tank (Blank). It is maybe because that the SDRAC–Water and SDRAC powder influence the coagulation process. The distillery sewage is acid wastewater and the suspended solids in the sewage can be regarded as suspended colloid particles in sewage. The charged colloid particle surface results in the repulsion of the same electrical property. The aggregation and precipitation fail. The activated carbon alkalified alkaline water and activated

**Table 2.** pH value change of SDRAC with different preparation conditions through the impregnation duration at 100 g: 200 mL of SDRAC: water

| Specimen | 1 h | 3 h | 5 h | 24 h |
|---|---|---|---|---|
| T750–60–200 [1] | 9.39 (0.16) [a 2)] | 9.50 (0.14) [a] | 9.60 (0.08) [a] | 9.58 (0.08) [a] |
| T800–60–200 | 9.47 (0.21) [a] | 9.58 (0.16) [a] | 9.67 (0.09) [a] | 9.64 (0.10) [a] |
| T850–60–200 | 9.40 (0.03) [a] | 9.48 (0.06) [a] | 9.49 (0.09) [a] | 9.48 (0.12) [a] |
| T850–90–200 | 9.64 (0.21) [a] | 9.6 4 (0.17) [a] | 9.57 (0.20) [a] | 9.54 (0.16) [a] |
| T850–90–500 | 10.09 (0.11) [b] | 9.97 (0.12) [ab] | 9.93 (0.09) [ab] | 9.80 (0.12) [a] |
| T750–60–200 (powder) [3] | 9.43 (0.03) [a] | 9.68 (0.11) [b] | 9.56 (0.08) [ab] | 9.54 (0.03) [ab] |
| T850–60–200 (powder) | 9.58(0.19) [a] | 9.67 (0.03) [a] | 9.52 (0.15) [a] | 9.57 (0.12) [a] |
| T850–90–500 (powder) | 9.53(0.04) [b] | 9.52 (0.06) [b] | 9.38 (0.03) [a] | 9.40 (0.02) [a] |
| SDR ash [4] | 11.23 (0.21) [a] | 11.06 (0.06) [a] | 11.02 (0.04) [a] | 10.98 (0.06) [a] |

[1) and 2)] are the same as Table 1
[3)] (powder) : SDRAC powder
[4)] SDR ash: SDR was prepared by combusted directly

**Table 3.** asic properties of distillery sewage from Kinmen Kaoliang liquor INC

| Sewage specimen from tank [1] | pH value | SS (mg/L) | EC (mS/cm) | COD (g/L) |
|---|---|---|---|---|
| Adjustment tank | 3.69 (0.02) [2] | 1963.33 (62.36) | 0.82 (0.01) | 98.02 (2.27) |
| Rapid mixing tank | 5.06 (0.61) | 2573.33 (83.80) | 2.73 (0.02) | 125.06 (2.73) |
| Floating tank | 5.40 (0.03) | 162.33 (5.44) | 2.58 (0.03) | 100.43 (2.62) |

[1)] Effluent: water specimen was taken from distillery sewage treatment after the second adjustment tank was sieved, the fast and slow mixing tank and the pressurized floating tank at 29th, Sept., 2016
[2)] See Table 1

carbon powder have the characteristics of alkali. They can neutralize the surface charge of the colloid to reduce the outer volume of the "Electric Double Layer" of the colloid surface. The suspended colloid is likely to agglomerate (Benefield *et al.*,1982).

**Improvement of distillery effluent into irrigation water**

*Analysis of physical properties*

1. Water temperature and DO

In the irrigation water standard (COA, 2003), the reproductive status of different crops is closely related to water temperature. For example, the yield decreases sharply when the rice water temperature is lower than 25°C (Taiwan organic information portal, 2008). The DO of general industrial wastewater are low and adverse for farmland irrigation. When the DO in water is too low, contaminants such as organics in water cannot be oxidized by biological metabolism. This often induces anaerobic soil as well as skin diseases for farmers when they work in the field. The irrigation water quality standard of Taiwan specifies that the DO is need to higher than 3 mg/L (COA, 2003; EPA, 2019). The Blank is an effluent specimen (Table 5). There was no significant difference between the water temperature and DO of the purified effluent. The effluent (Blank) according to Duncan's multiple range analysis and the irrigation water standard was met.

2. SS

The results of SS decreased by different purification methods with the SDRAC prepared in different conditions are also shown in Table 5. The SS of the Blank was 102.00 mg/L which exceeded the effluent standard (EPA, 2019). It decreased to 57.33–79.33 mg/L after filtration or standing. The standing method of T850 and the filtration and standing method of T800 had the best effect and the values were 24.00, 32.00, and 22.67 mg/L, respectively. The irrigation water standard specifies the SS in effluent at 50 and 100 mg/L. The effluent specimen treated by the SDRAC met the irrigation water quality standard (COA, 2003).

3. Oil and grease

According to the results of oil and grease decreased by different purification methods with the SDRAC prepared in different conditions, the oil and grease value of the effluent was 337.67 mg/L (Table 5). It exceeded the effluent standard (EPA, 2019) and the suspended matter in the original effluent results in large differences in the content of the water specimen. The standard deviation of the grease was large (247.71 mg/L). About 256–

**Table 4.** pH value, EC, SS and COD for sewage after simulation test

| Sewage specimen from tank [1)] | pH value | SS (mg/L) | EC (mS/cm) | COD (g/L) |
|---|---|---|---|---|
| Floating tank | 5.40 (0.03) [b 2)] | 162.33 (5.44) [a] | 2.58 (0.03) [b] | 100.43 (2.62) [a] |
| Blank [3)] | 3.14 (0.01) [a] | 933.33 (75.87) [c] | 1.43 (0.02) [a] | 96.39 (08.50) [a] |
| SDRAC–Water | 5.36 (0.03) [b] | 277.33 (19.14) [a] | 1.89 (0.00) [ab] | 110.16 (18.08) [a] |
| SDR Ash–Water | 5.11 (0.13) [b] | 941.33 (134.04) [c] | 2.40 (0.37) [b] | 158.36 (15.49) [ab] |
| NaOH | 5.12 (0.09) [b] | 980.00 (94.16) [c] | 3.90 (0.05) [c] | 130.41 (2.54) [ab] |
| SDRAC powder | 5.36 (0.26) [b] | 583.33 (181.54) [b] | 5.11 (0.71) [d] | 297.58 (184.05) [b] |

[1) and 2)] See Table 1, 3

[3)] Blank: sewage specimen from adjustment tank

**Table 5.** Physical properties of effluent after treating with different purifications by activated carbon, and for comparing with the effluent and the irrigation water standards

| Item [1)] | Water temperature (°C) | SS (mg/L) | Remove rate (%) [3)] | DO (mg/L) | Oil and grease (mg/L) | Remove rate (%) |
|---|---|---|---|---|---|---|
| Irrigation water standard | 38 | 100 | – | >3 | 5 | – |
| Effluent standard | 35 | 50 | – | – | 10 | – |
| Blank [2)] | 26 (0.47) [a] | 102.00 (4.32) [d 4)] | – | 7.97 (0.02) [bc] | 337.67 (247.71) [b] | – |
| T850–stand | 25 (0.47) [a] | 24.00(7.12) [a] | 76.4 | 7.73(0.10) [a] | 27.33(3.86) [a] | 91.9 |
| T850–filter | 26 (0.82) [a] | 44.67(3.77) [c] | 56.2 | 7.86(0.12) [ab] | 15.33(2.87) [a] | 95.4 |
| T850W–stand | 25 (0.82) [a] | 41.33 (1.89) [bc] | 59.4 | 8.02(0.05) [bc] | 38.67(29.10) [a] | 88.5 |
| T850W–filter | 26 (0.47) [a] | 40.67 (8.99) [bc] | 60.1 | 8.09(0.09) [c] | 78.33(6.85) [a] | 76.8 |
| T800–stand | 26 (0.47) [a] | 32.00 (2.83) [ab] | 68.6 | 7.94 (0.03) [bc] | 10.00(7.26) [a] | 97.0 |
| T800–filter | 26 (0.82) [a] | 22.67 (2.49) [a] | 77.7 | 7.98(0.03) [bc] | 81.00(38.76) [a] | 76.0 |

[1)] EPA, 2019, COA, 2003, and See Table 1; SDRAC–filtration method (stand; filter)

[2)] Effluent: water specimen was taken from distillery sewage treatment in Kinmen Kaoliang liquor INC. at 26th, March, 2017

[3)] Remove rate (%) = [(Blank–treated) /Blank] × 100

[4)] See Table 1 [2)]

327 mg/L removed by filtration or standing with a removal rate of 76–97%. The standard deviation (29.1 mg/L) was difference greatly. The effluent standard is 10 mg/L whereas the irrigation water standard is 5 mg/L (EPA, 2019; COA, 2003). The effluent after one standing or filtration treatment could not meet the water quality standard; therefore, the purified effluent was purified for the second time in this test. This results obtained that the T850–filter and T800–stand with the best effect were purified for the second time. The oil and grease value was decreased from 15 to 2.0 mg/L, that of the standing method was decreased to 10 mg/L, and met the water quality standard (Results no showed in Table).

*Analysis of chemical properties*

1. pH value

Table 6 shows the pH values of the effluent specimen after SDRAC filtration and standing. The pH value of Blank was 8.25 of slightly alkaline. The pH increased after purification (8.64–9.44). Therefore, the SDRAC filtration or standing increased the pH value. The effluent pH value specified by the agricultural irrigation water quality standard is 6.0–9.0 (COA, 2003). In terms of the water specimens treated by SDRAC, the water specimens treated by two purification methods with T850W met the water quality standard. There was a significant difference between T850W and the other conditions.

2. EC

As shown in Table 6, the EC of the Blank was 2.94. The EC increased after the SDRAC treatment. The increased EC value was 0.35–2.79 mS/cm. In addition, the EC increased after SDRAC filtration of different purification methods. The SDRAC filtration or standing method increased the results of EC. According to the electron potential theory in the alkaline solution environment, the positive valence metal tends to form a metal complex ($ROH \rightarrow R^{+}+OH^{-}$) with $OH^{-}$ in water. It is maybe because, in the course of adsorbing organic pollutants, activated carbon releases positive valence metal ions to the effluent by exchanging charges. This increases the EC value of the effluent. In addition, the activated carbon surface contains Al, Si, K, Ca, and Fe elements which increase the ions in water (Ahmedna *et al.*, 2004). It is suggested that the EC is increased as the result because the SDRAC is used, and its activation characteristics increase the EC value of water.

3. COD and BOD

The COD of effluent was 38.03 mg/L and the BOD was 5.18 mg/L. The values decreased after the SDRAC treatment. The COD and BOD decreased to 3.61–22.95 mg/L and 1.57–2.15 mg/L, respectively. The COD removal rate was 39.6–90.5% whereas the BOD removal rate was 58.4–69.6%. The T850 filtration method and T850W standing method had the highest COD removal rates (90.5 and 89.6%). There was no significant difference in the BOD of purified effluent. After the SDRAC filtration or standing treatment, the COD and BOD of effluent decreased, and met the irrigation water standard (COA, 2003).

4. Orthophosphate

The orthophosphate of Blank was 31.47 mg/L (Table 7). The orthophosphate value increased after the SDRAC filtration and standing (43.95–132.08 mg/L). The standing method had a higher orthophosphate value (64.98–132.08 mg/L) than the filtration method (43.95–91.54 mg/L).

5. $NO_3$–N, $NH_3$–N, and total nitrogen

In the effluent standard (EPA, 2019), nitrogen pollution includes sanitary problems such as water eutrophication, ecological balance, and bacterial growth. General inorganic nitrogen exists in seven oxidation states including $NH_3$, $N_2$, $N_2O$, $NO_2$, $N_2O_3$ ($NO_2^-$), $NO_2$, and $N_2O_5$ ($NO_3^-$). The effluent specification uses $NO_3$–N and $NH_3$–N as the sewage pollution indexes. The irrigation water quality standard uses total nitrogen to evaluate the feasibility of water irrigation (EPA, 2019; COA, 2003). Table 7 shows the $NO_3$–N, $NH_3$–N, and total nitrogen of the water specimen after the activated carbon filtration and standing. The $NO_3$–N, $NH_3$–N and total nitrogen values of effluent were 1.16 mg/L, 0.76 mg/L, and 1.22 mg/L, respectively. Their values after activated car-

**Table 6.** Chemical properties I of effluent after treating with different purifications by activated carbon, and for comparing with the effluent and the irrigation water standards

| Item I [1)] | pH value | EC (mS/cm) | COD (mg/L) | Remove rate (%) [3)] | BOD (mg/L) | Remove rate (%) |
|---|---|---|---|---|---|---|
| irrigation water standard | 6.0–9.0 | 0.75 | – | – | – | – |
| effluent standard | 6.0–9.0 | | 50 | – | 150 | – |
| Blank [2)] | 8.25 (0.03) [a 4)] | 2.94 (0.04) [a] | 38.03 (6.69) [c] | – | 5.18 (2.68) [b] | – |
| T850–filter | 9.18 (0.02) [c] | 4.64 (0.08) [d] | 03.61 (2.02) [a] | 90.5 | 1.83 (0.79) [a] | 64.6 |
| T850–stand | 9.44 (0.04) [e] | 5.73 (0.08) [f] | 08.85 (3.50) [a] | 76.7 | 2.01 (0.19) [a] | 61.1 |
| T850W–filter | 8.66 (0.03) [b] | 3.92 (0.04) [c] | 22.95 (2.82) [b] | 39.6 | 1.94 (0.22) [a] | 62.5 |
| T850W– stand | 8.64 (0.03) [b] | 4.49 (0.08) [d] | 03.93 (1.61) [a] | 89.6 | 1.57 (0.19) [a] | 69.6 |
| T800–filter | 9.14 (0.03) [c] | 3.29 (0.14) [b] | 08.85 (4.02) [a] | 76.7 | 2.15 (0.47) [a] | 58.4 |
| T800–stand | 9.31 (0.02) [d] | 3.96 (0.04) [c] | 11.48 (1.23) [a] | 69.8 | 1.77 (0.08) [a] | 65.8 |

[1), 2), 3) and 4)]: See Table 5

bon filtration and standing treatment were 0.03–0.37 mg/L, 0.60–0.76 mg/L, and 1.37–2.76 mg/L, respectively.

Nitrate ($NO_2^-$) is one of the forms of nitrogen dissolved in water. Other forms include $NH_3$–N, $NO_2$–N, and organic nitrogen (Org–N) (Sawyer, 1967). Various forms are converted into an aerobic state. In addition, activated carbon can adsorb onto the $NO_2$–N and $NO_2^-$ in water and decrease their contents to purify the water. (Mizuta *et al.*, 2004; Mishra and Patel, 2009). The T850 and T800 purification methods produced to remove $NH_3$–N. This is maybe because that the nitrogenous substance on the activated carbon surface is oxidized by nitrification to other forms of inorganic nitrogen e.g. $NO_2^-$ or $NH_3$–N. In terms of $NO_2^-$, the maximum removal rate of T850 filtration and standing treatment was 93.1–97.4%. The maximum $NH_3$–N removal rate of T850W filtration, standing, and T800 standing treatment was 13.1–21.1%. The $NO_2^-$, $NH_3$–N and total nitrogen values of the effluent treated by the SDR activated carbon filtration and standing can reach the irrigation water quality standard (COA, 2003).

6. Chloride

Table 8 shows the chloride value of effluent was 181.0 mg/L. After the activated carbon filtration and standing, the values of T850–stand and T850W–stand were 176.0 and 177.0 mg/L. These were slightly decreased. The values of T850W–filter and T800–stand increased to 184.0 and 196.0 mg/L, respectively. The chloride was difficult to be removed by adsorption because the chlorine has many ionic forms of different valences. It is difficult to be removed by single–form adsorption. The chloride is often regarded as one of the objects to be removed in the tertiary treatment of sewage. The removal methods include dilution method, precipitation method, ion exchange method, concentration by evaporation, coagulation and solvent extraction, electrolytic method, oxidizer method, and membrane separation (William, 1960).

7. Sulfate and sodium sulfate

Table 8 shows the sulfate values of the effluent after the activated carbon filtration and standing. None of the sulfate values were measured according to the detection method for sulfate in the water–turbidity method proclaimed by the EPA (2019). It is maybe because the effluent contains a lot of interfering organics. The barium sulfate cannot precipitate effectively in the test process. As the sulfate value cannot be measured, the sodium sulfate standard solution (100 mg/L) was prepared. The SDR activated carbon was used for filtration and standing treatment to investigate the effect of the SDR activated carbon on removing sulfate in water. It was able to remove 66–94% of sodium sulfate. The standing purification method has a better removal rate.

8. Anionic surfactant

Table 8 shows the value of effluent was 0.04 mg/L. The value after T850W standing is 0.04 mg/L. In terms of the SDR activated carbon standing test, the activated carbon treatment unincreased the anionic surfactant. The effluent and irrigation water standards (EPA, 2019; COA, 2003) are 10 and 5 mg/L. The treatment of this test was able to reach the standard for irrigation water.

*Analysis of metals and trace elements*

1. SDR and RSC

According to the sodium absorptivity results of effluent after activated carbon T850–90–500–Washed standing, the SAR of the effluent was 26.00 (√meq/L). The SAR value after standing was 29.80 (√meq/L). After the SDR activated carbon standing, the effluent SAR was increased by 3.8 (√meq/L), whereas the SAR specified by irrigation water quality standard is 6.0 (√meq/L) (COA, 2003). The SAR exceeded the standard. After activated carbon T850–90–500–Washed standing, the RSC of effluent was 0.632 (meq/L). The RSC value after standing was 0.340 (meq/L). Therefore, after the SDR activated carbon standing, the RSC value of residual sodium carbonate was decreased by 0.292 (meq/L). The removal rate was 46% which met the irrigation water standard (Results no showed in Table).

**Table 7.** Chemical properties Ⅱ of effluent water after been treated for different purifications by activated carbon, and for comparing with the effluent water and the irrigation water standards

| Item Ⅱ [1)] | Orthophosphate (mg/L) | $NO_3$–N (mg/L) | Remove rate (%) [3)] | $NH_3$–N (mg/L) | Remove rate (%) | Total nitrogen (mg/L) |
|---|---|---|---|---|---|---|
| irrigation water standard | – | – | – | – | – | 3.0 |
| effluent standard | 4.0 | 50 | – | 10 | – | – |
| Blank [2)] | 31.47 (0.01) [a 4)] | 1.16 (0.00) [g] | – | 0.76 (0.00) [b] | – | 1.22 (0.09) [a] |
| T850–filter | 88.64 (0.01) [d] | 0.03 (0.00) [a] | 97.4 | 0.75 (0.00) [b] | 01.3 | 1.37 (0.09) [a] |
| T850–stand | 118.03 (0.01) [f] | 0.08 (0.00) [b] | 93.1 | 0.76 (0.00) [b] | 00.0 | 1.68 (0.04) [b] |
| T850W–filter | 91.54 (0.00) [e] | 0.37 (0.00) [f] | 68.1 | 0.63 (0.00) [a] | 17.1 | 1.97 (0.16) [bc] |
| T850W–stand | 132.08 (0.00) [g] | 0.17 (0.00) [d] | 85.3 | 0.66 (0.00) [a] | 13.1 | 2.10 (0.06) [cd] |
| T800–filter | 43.95 (0.00) [b] | 0.34 (0.02) [e] | 70.6 | 0.75 (0.01) [b] | 01.3 | 2.34 (0.18) [d] |
| T800–stand | 64.98 (0.00) [c] | 0.13 (0.00) [c] | 88.7 | 0.60 (0.00) [a] | 21.0 | 2.76 (0.24) [f] |

1), 2), 3) and 4): See Table 5

2. Metals and trace elements

Table 9 shows the results of metals and trace elements of the distillery raw water, effluent, and purified effluent. The Zn, Cd, Ni, and Cu values of the distilled raw water were 0.1502, 0.0016, 0.0014, and 0.0075 ppm, respectively. After distillery distillation and raw sewage treatment, the Cr, Pb, Zn, Cd, Ni, and Cu values were 1.2, 0.0114, 0.0365, 0.0021, 0.0139, and 0.0208 ppm, respectively. The Cr, Pb, Ni, and Cu values were increased greatly. After T850–90–500–Washed standing treatment, the Cr and Pb values were lower than the detection limit. The removal rate was 99%. The Cr and Cu values were decreased after the SDR activated carbon purification, meeting the irrigation water quality standard (COA, 2003).

According to Table 9, the Be, Co, B, Mn, Al, and Li values of the distilled raw water were 0.0081, 0.0029, 0.014, 0.0338, 0.0204, and 0.0109 ppm, respectively. After distillery distillation and raw sewage treatment, the Be, Co, B, Mn, Al, and Li values were 0.0078, 0.0021, 0.0686, 0.3493, 0.0514, and 0.0132 ppm, respectively. The B, Mn, and Al values were increased greatly after the SDR activated carbon standing purification. The removal rate was able to be 99%. The Co, B, Mn, Al, and Li values were 0.0001, 0.0061, 0.0605, 0.0085, and 0.0091, respectively. The removal rate was 31–95%. Therefore, the items of common metal and trace elements meet the irrigation water quality standard (COA, 2003).

*Analysis of biological properties*

1. Total bacterial count and coliform group

The total bacterial count of the distillery effluent after the activated carbon standing was 55000 CFU/mL (Blank). The total bacterial count decreased signifi-

**Table 8.** Chemical properties Ⅲ of effluent water after been treated for different purifications by activated carbon, and for comparing with the effluent water and the irrigation water standard

| Item Ⅲ [1] | Chloride (mg/L) | Sulfate (mg/L) | Sodium sulfate (mg/L) | Remove rate (%) [3] | Anionic Surfactant (mg/L) |
|---|---|---|---|---|---|
| irrigation water standard | 175 | 200 | – | – | 5 |
| effluent standard | – | – | – | – | 10 |
| Blank [2] | 181.0 | N/D [4] | 100.0 | – | 0.04 |
| T850–filter | – | N/D | 11.1 | 88.8 | – |
| T850–stand | 176.0 | N/D | 10.7 | 89.2 | – |
| T850W–filter | 184.0 | N/D | 58.0 | 42.0 | – |
| T850W–stand | 177.0 | N/D | 5.4 | 94.6 | 0.04 |
| T800– filter | – | N/D | 99.86 | 0.0 | – |
| T800– stand | 196.0 | N/D | 33.8 | 66.2 | – |

[1), 2) and 3)]: See Table 5

[4] N/D: No Data

**Table 9.** Contents of metal and trace elements in original water, effluent water and water specimens for effluent after purification, and for comparing with the effluent water and the irrigation water standards

| Item [1] | Original water | Effluent | Effluent after purification | Remove rate (%) [2] | effluent water standard | irrigation water standard |
|---|---|---|---|---|---|---|
| Cr | N.D | 1.2 | N.D | 99.9 | 2.0 | 0.1 |
| Pb | N.D [3] | 0.0114 | N.D | 99.6 | 1.0 | 0.1 |
| Zn | 0.1502 | 0.0365 | 0.0044 | 87.9 | 5.0 | 2.0 |
| Cd | 0.0016 | 0.0021 | 0.0003 | 85.7 | 0.03 | 0.01 |
| Ni | 0.0014 | 0.0139 | 0.0035 | 74.8 | 1.0 | 0.2 |
| Cu | 0.0075 | 0.0208 | 0.0148 | 28.8 | 3.0 | 0.2 |
| Hg | N.D | N.D | N.D | – | 0.005 | 0.002 |
| As | N.D | N.D | 0.0094 | – | 0.5 | 0.05 |
| Be | 0.0081 | 0.0078 | N.D | 99.9 | – | 0.1 |
| Co | 0.0029 | 0.0021 | 0.0001 | 95.2 | – | 0.05 |
| B | 0.014 | 0.0686 | 0.0061 | 91.1 | 1.0 | 0.75 |
| Mn | 0.0338 | 0.3493 | 0.0605 | 83.7 | 10.0 | 0.2 |
| Al | 0.0204 | 0.0514 | 0.0085 | 83.5 | – | 5.0 |
| Li | 0.0109 | 0.0132 | 0.0091 | 31.1 | – | 2.5 |

[1), 2) and 3)]: See Table 5[1), 3) and 4] cantly after the activated carbon standing. The total bacterial count could be decreased by 98.2% to 40 CFU/ml. The purifying treatment after the activated carbon standing is an effective way to decrease the total bacterial count in water. The active mechanism presents a state of strongly alkaline solution in activated carbon. This eliminates the bacteria and the bacteria are adsorbed by activated carbon (Uraki *et al.*, 2008). Ogawa *et al.* (2011) indicated that the total bacterial count in water could be effectively decreased by the activated carbon standing. The coliform group of distillery effluent was 94.42 CFU/100 mL. After the activated carbon standing purification, the coliform group was 39.00 CFU/100 mL. This was a removal rate of 58.6%. The result shows that the coliform groups were decreased significantly after the SDR activated carbon standing (Results no showed in Table).

2. TOC

The changed in the TOC in water after the SDR activated carbon standing. The TOC of effluent was 57.9 mg/L. The TOC removal rate decreased from 32.4% to 39.1 mg/L after standing. The SDRAC was able to adsorb organic matter in water to reduce the content of TOC. The findings of the bioactive carbon filter of the Finland water purification plant show that the activated carbon filter treatment can remove 53–95% of natural organics from the raw water (Anu *et al.*, 2006). The findings of the SDRAC purified raw water from the Kinmen County reservoir and the slow filter water of water purification plants showed that the TOC removal rate of the SDRAC was 24.1–34.5%.

## CONCLUSION

The fermentation waste of SDR was prepared into SDR ash/SDRAC. It was then impregnated into the water to form an alkaline solution for the investigation on the feasibility of using it to treat distillery effluent. The prepared SDRAC yield was 19.11–28.91 and the iodine value was 261–774 mg/g. The iodine value of SDRAC prepared using activation temperature above 800°C, activation duration 90 min, and steam flow rate of 200 mL/h reached the commercial criterion. The pH of SDRAC increased with the activation temperature and steam flow rate, as well as with the SDRAC impregnation weight percent. According to ICP–OES, the SDRAC water contained Fe and Al metal ions. They formed a positive valence metal ion complex with water. Therefore, it is feasible to alkalify and settle sewage. The physical properties could be known from testing and then SDRAC was used to treat effluent based on the irrigation water standard, using this purification, 56–77% of SS and 76–97% of oil and grease was able to be removed. The chemical properties of COD, BOD, $NO_3$–N, and $NH_3$–N decreased 39–90, 58–65, 68–97 and 1–21%, respectively. The metals and trace elements in water removed 28–99 and 31–99%, respectively. The total bacterial and coliform count of the water specimen after purification decreased about 98.2 and 58.6%, respectively. TOC in water decreased about 32.4%. In summary, the SDRAC is impregnated into the water to form alkaline water. It is feasible to alkalify and settle sewage. The SDRAC from the precursor SDR after impregnation has the applied value to purify the distillery effluent into irrigation water.

## AUTHOR CONTRIBUTION

Kun–You Li performed the course/experiments and evaluated data with the statistical analysis. Noboru Fujimoto supervised the work. Han Chien Lin designed the study and wrote this paper. The authors assisted in editing of the manuscript and approved the final version.

## ACKNOWLEDGEMENTS

The authors offer our sincere appreciation to the Ministry of Science and Technology, Taiwan ROC through Grant No. MOST 105–2633–E–415 –001 – for financial support.

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