World Journal of Analytical Chemistry, 2025, Vol. 10, No. 2, 37-47
Available online at http://pubs.sciepub.com/wjac/10/2/1
Published by Science and Education Publishing
DOI:10.12691/wjac-10-2-1

Use of Membrane Technologies for the Stabilization of Hibiscus Sabdariffa L. Extract: New Electrochemical Approach

Ndiaye Khady*, Kane Cheikhou, Ayessou Nicolas, Cisse Mady, Diop Codou Mar

Laboratory Water Energy Environment and Industrial Processes, ESP, UCAD, Sénégal
*Corresponding author: khadyameth@yahoo.fr

Received September 20, 2025; Revised October 22, 2025; Accepted October 29, 2025

Abstract The instability of anthocyanins, the molecules responsible for the red coloration of *Hibiscus sabdariffa L* calyxes, remains a major problem despite the many stabilization techniques that exist. Membrane technologies have played a central role in stabilizing roselle anthocyanins. Until now, dissolved oxygen in juices has been treated by bubbling with an inert gas or by adding other molecules such as preservatives, which can cause considerable damage to human health. The electrochemical approach is a new stabilization technique that reduces dissolved oxygen in juice, cold and without adding other molecules. The reduction of dissolved oxygen on a platinum electrode is a new athermic technique using an electrolysis cell with two compartments separated by a cationic membrane. The fruit juice is stabilized by passing a reduction current through it for a specified period of time. The results obtained from bubbling reveal that it is necessary to bubble with nitrogen gas for 2 hours to obtain an anthocyanin concentration of 279.21 mg/L after storage at 37°C for 30 days, while 282.66 mg/L is obtained for the same extract treated electrochemically (30 min/-6 mA) and stored under the same conditions. Both concentrations are significantly different from the control concentration of 263.37 mg/L. Electrochemical treatment with a time/current combination of 30 min/-6 mA for 500 ml of *Hibiscus sabdariffa L* extract on a platinum/SCE electrode (in a batch reactor) preserved more than 10% of the anthocyanins at 25°C and 37°C after 30 days of storage, which is significantly more than the control. This had previously been problematic for other treatments. The classic Arrhenius, Ball, and Eyring models used showed the degrading effect of dissolved oxygen in the extract, with a significant difference (Ea = 4000 J/mol) between the activation energy of the electrically reduced extract and the control (untreated). This was corroborated by half-reaction time values of 24 s^{-1} for the electrochemically treated extract and 20 s^{-1} for the untreated control after 5 months of storage at 4°C. The activation enthalpy of the electrically reduced hibiscus extract is approximately 6 J/mol/K, and that of the control is 5.72 J/mol/K, showing that the electrochemical process occurs at low energy, i.e., 0.28 J/mol/K for a volume of approximately 250 ml.

Keywords: *Membrane, Stabilization, Anthocyanin, Oxygen, Electrochemistry, Hibiscus*

Cite This Article: Ndiaye Khady, Kane Cheikhou, Ayessou Nicolas, Cisse Mady, and Diop Codou Mar, "Use of Membrane Technologies for the Stabilization of Hibiscus Sabdariffa L. Extract: New Electrochemical Approach." *World Journal of Analytical Chemistry*, vol. 10, no. 2 (2025): 37-47. doi: 10.12691/wjac-10-2-1.

1. Introduction

Hibiscus sabdariffa L., commonly known as bissap, karkadé, ngai ngai, roselle, among others, is a herbaceous plant belonging to the Malvaceae family. Hibiscus is cultivated in tropical regions such as Senegal. Anthocyanins are polyphenolic compounds responsible for the red color of roselle calyxes. These molecules are rich in antioxidants and have proven health benefits. However, anthocyanins are unstable because they are sensitive to heat, light, pH, and oxygen, which cause them to degrade and lose their color [1,2,3,4].

Several methods have been explored to improve the stability of anthocyanins in *Hibiscus sabdariffa L* extracts:

Extraction methods aimed at improving anthocyanin stability and yield were developed by Hoang Thi Ngoc Nhon et al. [5]. A combination of enzyme-assisted and microwave extraction with co-pigments such as catechin, chitosan, and chondroitin sulfate extended the shelf life of anthocyanin powder by six months.

Cold extraction has been shown to preserve anthocyanin content more effectively. Cold extraction processes reduce anthocyanin degradation during storage, thus preserving the color and nutritional quality of the extracts over time [6].

The heat sensitivity of anthocyanins in *Hibiscus sabdariffa L.* has been the subject of numerous studies, highlighting the significant impact of temperature on their stability. Research indicates that anthocyanins degrade more rapidly at high temperatures, particularly during hot extraction and pasteurization processes. For example, a study conducted by Cissé et al [7] demonstrated that

extracts subjected to pasteurization at 100°C for 5 minutes suffered a loss of 20 mg/L of anthocyanins, while those extracted at 100°C for 240 minutes showed greater degradation, highlighting the adverse effects of prolonged exposure to heat on anthocyanin content.

These advances in extraction and stabilization techniques offer promising prospects for preserving the vivid color and beneficial properties of anthocyanins from *Hibiscus sabdariffa L.*

Cissé and his colleagues conducted research on the stabilization of anthocyanins in extracts of *Hibiscus sabdariffa L.*, focusing on athermal techniques that avoid the degradation associated with heat treatment.

Cissé showed in these studies that tangential microfiltration using ceramic membranes allows roselle extracts to be concentrated without significant microbial growth for 90 days at temperatures of 4°C, 20°C, and 37°C. However, losses in anthocyanins and vitamin C were observed during storage [7].

Athermal concentration can be achieved by osmotic evaporation. In pilot studies, roselle extracts were concentrated using hydrophobic polypropylene hollow fiber membranes. This process achieved a total soluble solids (TSS) content of 610 g/kg at 45°C, with an evaporation flux of 1.5 kg/h·m². Anthocyanins and vitamin C are effectively preserved compared to traditional thermal methods [8].

A method for removing radical and molecular oxygen using three enzymes: an oxidase and its substrate, catalase, and superoxide dismutase is carried out [9].

Sugawara et al. have patented a process for producing liquid milk concentrate and powdered milk, combining the removal of ions from milk and the reduction of dissolved oxygen, followed by thermal sterilization, thereby improving the physicochemical and organoleptic properties of the food raw material. After ion removal, the milk is brought into contact with an inert gas.

However, modifying the basic components of food by adding other products is problematic. The instability of anthocyanins, the molecules responsible for the red coloration of *Hibiscus sabdariffa* calyxes, remains problematic despite numerous stabilization techniques on the subject.

Innovations in preservation are increasingly focused on developing more efficient processes and technologies tailored to specific needs. One such advance is the electrochemical reduction of dissolved oxygen using a platinum electrode in an electrolysis cell with two compartments separated by a cationic membrane. This athermal technique offers an innovative approach to stabilizing fruit juices, such as *Hibiscus sabdariffa L.* extract, by reducing oxidative degradation without the use of heat or chemical additives.

The electrochemical method involves applying a reduction current through a platinum electrode, effectively removing dissolved oxygen from the extract. This process preserves the organoleptic qualities of the product and extends its shelf life. This approach was first used in the preservation of hibiscus extract [10], demonstrating its potential as an alternative to traditional methods such as nitrogen gas bubbling or enzymatic oxygen removal.

This electrochemical treatment, applied for 30 minutes at -6 mA, resulted in 10% higher anthocyanin preservation compared to the untreated control after 30 days of storage at 25°C and 37°C [11].

These studies highlight the potential of membrane and electrochemical methods for preserving the stability of anthocyanins in *Hibiscus sabdariffa L.* extracts, thus offering alternatives to conventional heat treatments that can degrade these sensitive compounds.

2. Hibiscus Sadariffa

Hibiscus sabdariffa L is a herbaceous plant in the Malvaceae family.

There are two varieties of *Hibiscus sabdariffa*: Hibiscus sabdariffa variety altissima and *Hibiscus sabdariffa L* [9]. These plants have red or green calyxes. *Hibiscus sabdariffa L* is a vigorous, bushy, vascular plant in the Malvaceae family, with edible calyxes [5,9]. The calyx is made up of five sepals, joined by a more or less dark-red outer calicule, which can be shaded from red to green on the inside. The fruit has five chambers and contains around twenty black seeds with an oily albumen [1].

Figure 1. Hibiscus sabdariffa L. calyx

2.1. Biochemical and Mineral Compositions

Fresh calyxes contain biochemical molecules such as carbohydrates, lipids and proteins. Calyxes are also rich in minerals, including iron, calcium, phosphorus, magnesium, zinc and potassium. The content of certain elements in the calyx varies according to crop, origin, soil, rainfall and variety [2], as shown in Table 1 below.

Table 1. Minimum, average and maximum biomolecule and mineral values for fresh calyxes of Hibiscus sabdariffa L. [2]

Type de données	Humidité	Protéines	Lipides	Fibres	Cendres	Glucides	Acide malique	Calcium	Fer	Phosphore	Acide ascorbique	Anthocyanes
	(g/100g)							(mg/100g)				
Minimales	84 ,5	0 ,9	0,1	2,5	4,5	3,3	0,12	1,3	2,9	40,0	6,7	150
Moyenne	86,3	6,6	2,3	8,8	5,6	8,1	1,36	94,0	17, 2	191,1	72,0	350
Maximales	89,5	17 ,9	3 ,9	12,0	6 ,8	12,3	2,70	213,0	37, 8	312,6	141,1	1500

2.2. Anthocyanins

These are polyphenols, compounds with an aromatic nucleus containing one or more hydroxyl substituents and derived functional groups. Anthocyanins belong to the flavonoid family, pigments responsible for the coloring of plant flowers, fruits and leaves [3].

The precursors of anthocyanins are flavanones. The aglycone of anthocyanins is called anthocyanidin: it is the flavilium cation [4].

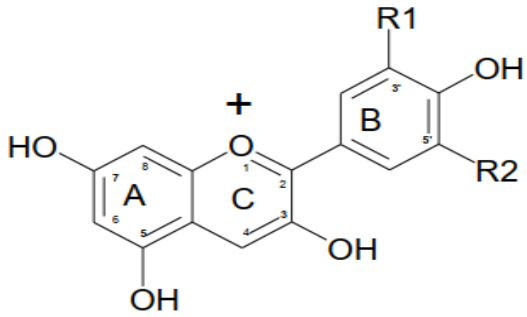

Figure 2. Flavylium/2-phenyl-1-benzopyrilium cation [5]

The anthocyanins most commonly found in plants are shown in Table 2.

Table 2. Main anthocyanins in plants [6]

Anthocyanidin	R1	R2	Color at pH1
Delphinidin	OH	OH	Blue-violet
Cyanidin	OH	H	Red
Pelargonidin	H	H	Orange
Petunidin	OCH_3	OH	Blue-purple
Peonidin	OCH_3	H	Pink-red
Malvidin	OCH_3	OCH_3	Purple

2.3. The Therapeutic Properties of *Hibiscus sabdariffa L.*

The calyxes of *Hibiscus sabdariffa L* are rich in anthocyanins, responsible for its red color. These anthocyanins possess a number of therapeutic virtues that have been the subject of numerous research projects.

2.3.1. Anti-hypertensive

The use of *Hibiscus sabadariffa L* extract is considered in traditional Chinese medicine as an alternative or complementary medicine to the treatment of certain diseases such as hypertension and cancer, and as an antibacterial agent.

K. PURO et al presented in a mini review: the medicinal uses of the Hibiscus sabdariffa plant. The review compiles several results on the therapeutic properties of Hibiscus extract.

One study showed a significant reduction in both systolic and diastolic blood pressure. Results from studies on rats confirmed that Hibiscus sabdariffa extract contains antihypertensive constituents [7].

2.3.2. Antibacterial

Studies on Hibiscus extract have demonstrated the presence of phytochemical constituents, cytotoxic and antimicrobial activities. The extract showed antibacterial activity against *Staphylococcus aureus, Bacillus stereothermophilus, micrococcus luteus, Serratia mascences, Clostridium sporogenes, Escherichia coli. Klebsiella, Pneumoniae, Bacillus cereus, Pseudomonas fluorescence* [7]. These results are corroborated by those of Ines Da-costa-Rocha et al [8] in a phytochemical and pharmacological review.

2.3.3. Anti-carcinogenic

The anti-carcinogenic properties of the Hibiscus sabdariffa plant were tested on ovarian (MDA-MB-231), breast (MCF-7) and cervical (HeLa) cancer cell lines. Results showed the highest antiproliferative potency against breast cancer cells (MCF-7) [8]. These results are supported by those of Bo-Wen Lin [9] in his review article published in the British Journal of Pharmacology and Daotong Li [10] in his critical review published in Food Science and Nutrition.

Another author, Laércio Galvao Maciel [11], has also shown that anthocyanins possess high antioxidant (DPPH) and biological (tumour cell lines) activity.

2.3.4. Anti-inflammatory

Ines Da-costa-Rocha in a review published in food Chemistry, presented clinical studies on ten volunteers who showed a decrease in plasma concentration of monocyte chemoattractant protein 1 (MCP-1). This result is confirmed by those of R. Beltran-Debon [12].

Hibiscus extracts have also been implicated in the inhibition of certain substances, resulting in an anti-inflammatory effect [8].

2.3.5. Anti-anaemia

The rich mineral content of the calyxes [13] explains why Hibiscus juice can help combat certain diseases linked to a deficiency of minerals such as iron.

Preliminary studies [14] have shown that the use of a decoction of *Hibiscus sabdariffa L* calyxes can be an alternative treatment for anemia.

2.4. Anthocyanin Degradation Factors

Storage processes usually lead to a reduction in flavonoid content. This reduction depends on storage time and temperature, the nature of the flavonoids and the food matrix stored.

Light, storage time and oxygen have an effect on the stability of flavonoids in solution and on the evolution of their antioxidant activity [15].

2.4.1. Hydrogen Potential (pH)

In a strong acidic aqueous medium, anthocyanins are in their ionic forms, with the red-colored flavylium cation being the dominant form [4].

In weak acidic conditions, the cation loses its proton, leading to the formation of a blue quinone base. Its hydration leads to a colorless hemiacetal and then, by tautomerism, to a chalcone form [4].

Polyphenols in fruit and vegetables are more stable the lower the pH value. The effect of pH has an important

influence on flavonoid degradation [16].

Flavonoids are unstable in aqueous media, and the main degradation reactions involve hydroxylation, oxidation and cyclic cleavage.

The evolution of vitamin C content as a function of time (20 days) and pH has shown a decrease in ascorbic acid with increasing pH [17].

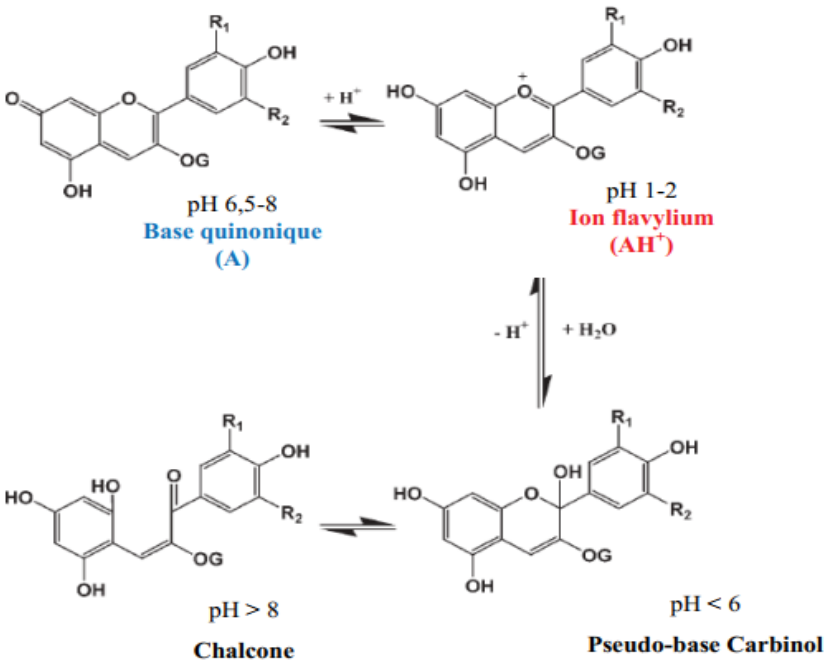

Figure 3. Main forms of anthocyanins in aqueous media [7]

2.4.2. Temperature

Increasing temperature leads to the opening of the heterocycle, resulting in the formation of colorless products [18].

The degrading effect of temperature on anthocyanin extraction was characterized by M. CISSE [19]. These results show that at room temperature (25°C) the concentration of extracted anthocyanins is optimal, compared with higher temperatures where a considerable reduction in anthocyanin concentration is observed.

2.4.3. Light

Light has an impact on anthocyanin stability. Siti NURYANTI et al [20] carried out tests on cyanidin-3-glycoside in the presence and absence of light. The results showed that light has an impact on anthocyanin stability. In vitro in extracts, light accelerates anthocyanin degradation [4].

2.4.4. Metal ions

Transition metals such as iron, tin, copper, zinc, magnesium etc. complex with anthocyanins, leading to their degradation. The anthocyanin degradation reaction is thought to be catalyzed by metal ions [18].

A study of the effect of metal ions on the thermal degradation of quercetin indicated that reactive oxygen species may explain the increased lability of flavonol in aqueous solution [21].

2.4.5. Oxygen

The term dissolved oxygen is commonly used for "dissolved oxygen". This measurement refers exclusively to molecular oxygen (O_2).

Dissolved oxygen is essential for most biological processes that support life, both on land and in the sea. The concentration of dissolved oxygen in water is the result of physical (temperature, salinity, mixing of the water mass), chemical and biological parameters. In its absence, or below certain concentrations, consequences can lead to the death of living species [22].

Oxygen is present in water in the form of gaseous molecules, within tiny air bubbles. It dissolves in water by diffusion, until an equilibrium called saturation is reached [23].

Deoxygenation is often carried out using a neutral gas to reduce the concentration of dissolved oxygen. However, this technique also leads to organoleptic impoverishment of the wine, so it should only be used in exceptional cases.

Dissolved oxygen has an impact on the sensory qualities of food products [18,21,22,23,24,25].

The action of dissolved oxygen on anthocyanins is generally facilitated by the presence of minerals [26].

The increased instability of flavonols in aqueous solutions is due to reactive oxygen species [21]. According to the latter, quercetin and morin were oxidized by hydroxyl free radicals (HFR).

According to Cejudo et al, [27] anthocyanin-rich, micro-oxygenated red wines showed significantly different chromatic characteristics from non-oxygenated wines, with the micro-oxygenated wines recording the lowest chroma values. They showed that tannins, flavonols, anthocyanins and flavan-3-ol in microoxygenated wine decreased after 5 months' storage compared with non-microoxygenated wine. But anthocyanin-bound red pigments were the main phenolic compounds significantly affected by the microoxygenation treatment.

They justified [27] the low flavan-3-ol content after oxygen addition by the fact that oxygen activates reactions between free anthocyanins and flavan-3-ol, thus producing new anthocyanin-derived pigments [28].

Boido et al, noted a decrease in the red color component (a*) and an increase in the yellow component (b*) for the wine studied after 5 months' storage.

Both aerobic and anaerobic vitamin C degradation pathways may be involved in non-enzymatic browning attributed to Maillard reactions. Intermediate products of non-enzymatic browning may polymerize or combine with amino acids to give brown melanin pigments such as furfural and 2-furoic acid. The presence of citric acid can also promote browning. Ascorbic acid oxidation is promoted by temperature, metal ions and dissolved oxygen.

Self-oxidation of ascorbic acid has been shown to strongly degrade tea samples [17].

Oxygen is also involved in the first stage of enzymatic browning reactions (enzymatic oxidation), giving o-quinones followed by condensation reactions [29].

3. Membrane Techniques for Stabilizing *hibiscus sabdariffa l.* Extract

A process for extracting and concentrating anthocyanins from Hibiscus rosa-sinensis was developed by Atreyee and colleagues [30].

A maximum anthocyanin content of 461.39 mg/kg was obtained using: a citrate-phosphate buffer at pH 4.6, a liquid/solid ratio of 20:1, and an extraction time of 4 hours. Concentration by forced osmosis of this content (461.39 mg/kg) resulted in an anthocyanin content of 3967.9 mg/kg.

Concentration by forced osmosis had no impact on the total content of anthocyanins, phenolic compounds, antioxidant activities, or color parameters.

The process diagram is as follows

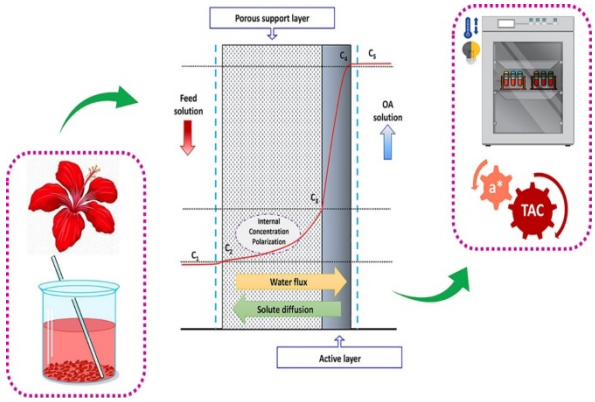

Figure 4. Schematic diagram of the forced osmosis extraction process [30]

These results corroborated those of **Cissé et al**, who tested ten flat sheet nanofiltration membranes and eight sealed ultrafiltration membranes with nominal molecular weight cut-off (MWCO) values ranging from 0.2 to 150 kDa. They obtained a significantly higher anthocyanin content for the nanofiltration membranes [31].

In addition, 100% anthocyanin retention was noted for one of the membranes tested on an industrial scale. A filtration area of 2.5 m² made it possible to concentrate the roselle extract from 4 to 25 g of total soluble solids per 100 g.

In the same vein, **Adje Anoh Félix et al** [32] developed an eco-extraction process, comprising coupled technologies (tangential microfiltration: TFM and reverse osmosis: RO), which enabled the pilot-scale production of concentrated extracts with greater safety (quality and hygiene) by evaluating the stability of the concentrated extracts.

The polyphenolic extracts were clarified by Tangential Microfiltration (TMF) using an industrial multi-channel membrane (P19-60, pore diameter 0.2 µm, S = 0.304 m^2 filter surface area), then concentrated by RO using an industrial membrane (SW-30, pore diameter 0.1 nm, S = 2 m^2). These results guarantee microbiological stability and thus the availability of stable Hibiscus extract throughout the year.

Figure 5. Membrane Technology Pilot Scale

However, since fouling is a limitation of membrane technologies, **Carolina Moser Paraíso et al [33]** used a turbulence promoter and ultrasound to mitigate fouling during the ultrafiltration of hibiscus extract.

The use of a turbulence promoter and ultrasound for the treatment of hibiscus extract with a 5 kDa membrane resulted in a considerable increase in steady-state flow from 97.55 ± 0.05 to 367.61 ± 0.74 kg $h^{-1}m^{-2}$.

More than 60% of the main compounds (total phenolic compounds, total anthocyanins, cyanidin-3-glucoside, delphinidin, quercetin, myricetin, and rutin) in the Hibiscus extract were retained by the membrane, showing that the quality of the product is not altered by the application of turbulence promoter and ultrasound.

The process diagram is shown in Figure 6.

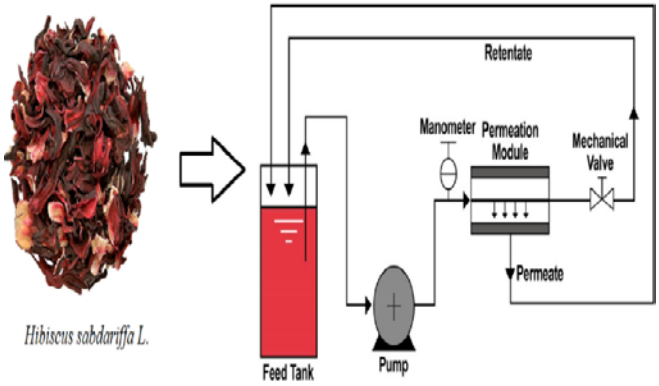

Figure 6. Schematic diagram [33]

In another study, **Carolina Moser Paraíso et al**. [33] evaluated a filtration process with and without centrifugation through asymmetric hollow spinel membranes for the clarification of hibiscus extract.

Sequential membrane filtration reduced total soluble solids from 5.53 to 4.03 °Brix, total solids from 47,120 to 37,780 mg/L, and the L* color coordinate (lightness) from 15.69 to 22.03.

After 20 days of storage, the filtered hibiscus extract showed greater stability compared to the food extract, with a 43.1% reduction in tea cream formation.

The combination of sequential centrifugation and filtration using asymmetric spinel hollow fiber membranes is a method that allows for the production of antioxidant-rich hibiscus tea.

According to **Cissé et al.** [34], the stability of roselle extract at low temperatures through tangential microfiltration is possible by setting the optimal parameters based on the VRR:

- For a volumetric reduction ratio (VRR) of 1, the optimal transmembrane pressure was 2.5 bar and the permeate flow rate was 185 L h^{-1} m^{-2}.
- For a volumetric reduction ratio (VRR) of 20, the optimal pressure was 3.7 bar with a permeate flow rate of 95 L h^{-1} m^{-2}.

Hibiscus extract treated by tangential microfiltration and stored at 4 and 20°C remained stable after 90 days of storage. However, at 37°C, product stability remains problematic.

Thi Ngoc Nhon Hoang et al. [35] used a D101 macroporous resin to improve the purity of anthocyanins in *Hibiscus sabdariffa L*. extract.

The resin enabled purification of the anthocyanin fraction into its main component (cyanidin-3-O-sambubioside) at 72.18% compared to 12.18% for the unpurified extract.

However, the good color stability of the purified anthocyanins is noted at low temperatures and in a slightly alkaline environment.

The basic diagram is as follows (Figure 7):

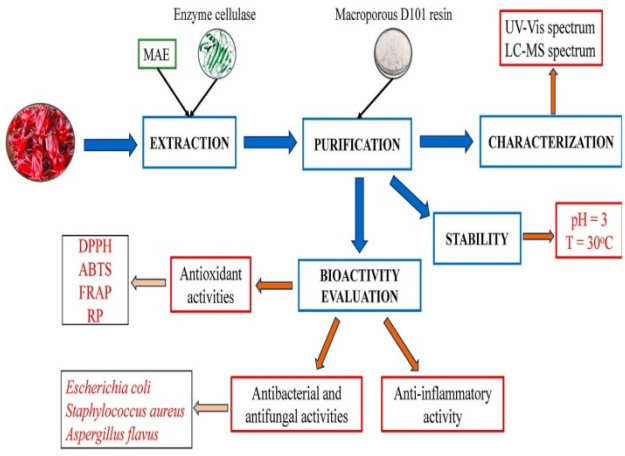

Figure 7. Process diagram [35]

Scientific literature has shown that Hibiscus extract can be stabilized using a membrane process for storage at low temperatures. However, at 37°C, the temperature factor seems to predominate.

4. Techniques for Stabilizing *Hibiscus sabdariffa l.* extract by Electrochemical Means

This is a new method using a commercial cationic membrane of the nafion type. The treatment of Hibiscus sabdariffa extract was carried out by electrochemical reduction of dissolved oxygen.

The electrochemical measurements carried out in this work use a potentiostatic set-up with three electrodes. It comprises a platinum working electrode, the site of the electrochemical reactions studied, a stainless-steel auxiliary electrode which closes the electrical circuit and a saturated calomel reference electrode, which makes it possible to control and measure the potential of the working electrode at each moment. The system used is a potentiostat connected to a computer equipped with cyclic voltammetry software (Figure 8) [36].

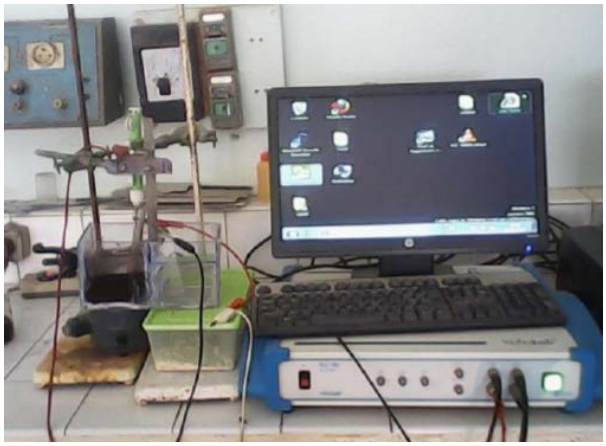

Figure 8. Electrochemical device for the electroreduction of *Hibiscus sabdariffa L* juice

A Nafion-type cationic membrane was used in the construction of the Plexiglas electrolysis cell. The cell has two compartments separated by the cationic membrane. One compartment of the electrochemical cell contains the Hibiscus extract to be treated and the other compartment contains a 0.1N hydrochloric acid solution.

A PGZ 100 potentiostat defines the potential between the cathode and the reference electrode [36].

The potential scan was performed between -0.5V and 1.5V/ECS at a rate of 100mV/s.

At the cathode, oxygen is reduced on the platinum electrode according to the acid reaction

$$O_2 + 4H^+ + 4e^- \rightarrow 2H_2O \quad (1)$$

The reduction of one oxygen molecule into two water molecules on the platinum electrode occurs in particular via a 4-electron process [37].

At the end of each electrolysis, the treated product is placed in small bottles wrapped in aluminium foil for storage at 4°C, 25°C and 37°C. Anthocyanins and colour were monitored every month.

4.1. Results and Discussion

4.1.1. Electrochemical Characterization

In this section, the author [36] performed cyclic voltammetry characterization of dissolved oxygen, the treatment time/reduction current intensity pair, and the optimal electrode surface area for the electroreduction of roselle extract.

Figure 9 below shows the voltamograms of the hibiscus extract before and after electroreduction, respectively (Figure 9A and Figure 9A'), and the voltamograms of distilled water (blank) before and after electroreduction (Figure 9B and Figure 9B').

Electroanalysis by cyclic voltammetry of the extract and blank before reduction of dissolved oxygen shows peaks at -5 mA and -13 mA (respectively). After electroreduction by chronopotentiometry, cyclic voltammetry of the extract and blank showed peaks reduced by -2.5 mA and -6.5 mA, respectively. This could be explained by the reduction of dissolved oxygen in solution. Electrolysis of the blank (distilled water) revealed that the electroactive element present in the hibiscus extract is the same as that present in the blank: dissolved oxygen.

In order to confirm the degrading effect of dissolved oxygen in the roselle extract, the author bubbled extracts of the same volume with inert gas in 30-minute increments. The bubbled samples were then stored at 37°C for 4 weeks. The results obtained are presented in Table 3 below.

The concentration of hibiscus anthocyanins was well preserved in the bubbled samples compared to the non-bubbled control. The extract that was bubbled for more than 2 hours had the highest anthocyanin concentration (279.21 mg/L) compared to the extract bubbled for 30 minutes (250.30 mg/L). Therefore, dissolved oxygen in roselle extract is a factor in the degradation of anthocyanins. However, on an industrial scale, the quantity of inert gas required for large volumes remains problematic.

Characterization of the electrode surface led to the decision to immerse the electrodes 4 cm into the solution for electrochemical treatment of the extract. The results are shown in the following table (Table 4).

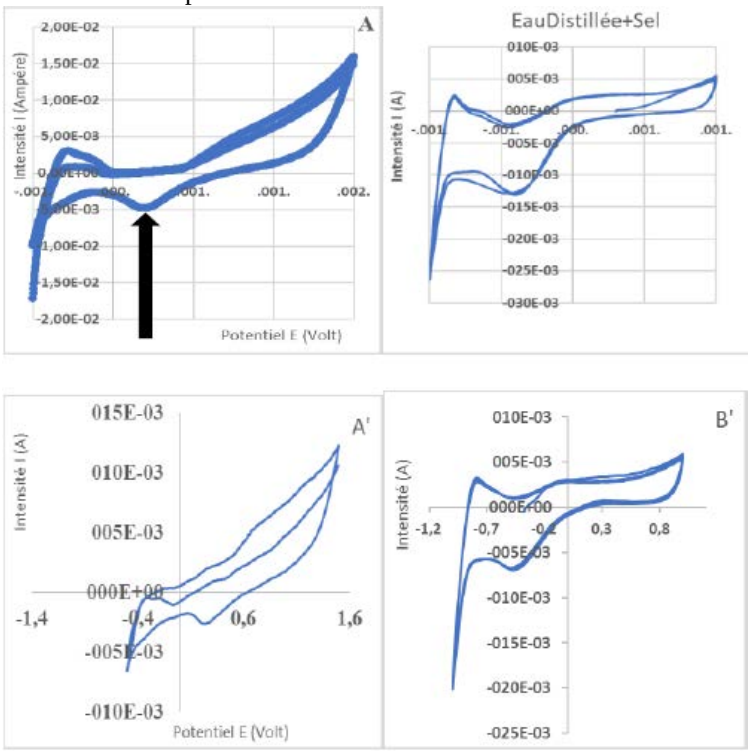

Figure 9. A-Voltammogram of Hibiscus sabdariffa extract; A'-Voltammogram of electroreduced Hibiscus sabdariffa extract; B-Voltammogram of the blank; B'-Voltammogram of the electroreduced blank [36]

Table 3. Dissolved oxygen and anthocyanin concentration (after one month of storage at 37°C) of Hibiscus sabdariffa L extract bubbled with nitrogen gas [36]

Bubbling time	Initial	30min	1hour	1h30min	2hours	2h30min
[O_2] (mg/L)	8	5,62	3,15	2,37	2,11	1,53
[Ant] (mg/L)	393,33$^{(a)}$	250,30$^{(b)}$	256,02$^{(bc)}$	264,29$^{(c)}$	268,88$^{(dc)}$	279,21$^{(d)}$

Table 4. Anthocyanin concentration of electroreduced extracts having the platinum plate as working electrode [36]

Time/intensity	Anthocyanin Concentration (mg/L)	% loss	Standard deviation
30-10_4cm	260,38$^{(ab)}$	33,80	5,53
40-6_4cm	272,09$^{(a)}$	30,82	9,36
30-6_4cm	282,66$^{(a)}$	28,14	11,38
Control	245,00$^{(b)}$	37,71	12,00

The results in Table 4 show a non-significant difference in the anthocyanin concentration of the samples electroreduced with the 30mn/-10mA_4cm pair and the control. However, there is a significant difference in the concentration of the pairs: 40mn/-6mA_4cm; 30mn/-6mA_4cm and the control.

The stabilization treatment of the extracts (500 mL) can be carried out for 30 minutes at a current intensity of -6 mA (282.66 mg/L).

4.1.2. Monitoring of Anthocyanins in Hibiscus Extract after Electro-reduction

In this section, the author [38] monitored the anthocyanins and color of *Hibiscus sabdariffa* extract. The results obtained showed significant differences between the electrically reduced extract and the untreated control.

The reduction of dissolved oxygen in 200 mL of Hibiscus extract was carried out at a potential of -250 mV for 15 minutes. The calyx/water ratio was 1/15. After electroreduction, the extracts were stored at 4°C and 37°C, and the anthocyanins were monitored during the first and sixth months.

The Figure 10 shows the results obtained.

The results (Figure 10) showed a significant difference in anthocyanin concentration between the extract with reduced oxygen and the control sample without electrically reduced oxygen during the first and sixth months of storage at 4°C and 37°C.

A difference of more than 5% between the sample with electrically reduced oxygen and the untreated control was observed in the first month of storage at 4°C and 37°C. After six months of storage, a 3% difference was noted between the sample and the control stored at 37°C.

Electrochemical treatment can therefore be an alternative to bubbling.

Tests to stabilize Hibiscus anthocyanins using membrane technologies have shown their limitations at high storage temperatures [34].

Furthermore, color monitoring, particularly using cielab coordinates, corroborated the results obtained with anthocyanin monitoring.

Color monitoring corroborates anthocyanin monitoring. The results for color parameters are presented in the following table, shows the red/green a* ratio of the sample and the control after storage at 4°C and 37°C.

Table 5. Red/green color component (a*) of electroreduced (sample) and non-electroreduced (control) extract stored at 4°C and 37°C [38]

	Red/green component a*			
			1month	6months
4°C	Sample		50,09[a]	48,63[b]
	Control		49,39b	47,53[c]
37°C	Sample		48,56[a]	45,36[c]
	Control		47,96[b]	44,18[d]

The red component of the electro-reduced extract retains a significant difference between the untreated control and the electro-reduced extract at 4°C and 37°C after 1 month and 6 months of storage (Table 5).

The reduction of dissolved oxygen in the Hibiscus extract allowed for good preservation of the anthocyanins in *Hibiscus sabdariffa L.*, which are responsible for the red color of roselle.

The author then compared the two methods, chronopotentiometry and chronoamperometry.

Two electrochemical methods can be used to reduce oxygen in roselle extract. These are chronopotentiometry (fixing the intensity for a given time) and chronoamperometry, which involves applying a potential for a certain period of time.

Figure 11 shows the monitoring of the anthocyanin concentration of the electroreduced extract and the control after 5 months of storage at 4°C, 30°C and 37°C.

Based on the results of the experiment (figure), the two electrochemical methods appear to be equivalent. Nevertheless, some insignificant differences were noted.

The reduction of dissolved oxygen in the Hibiscus extract can be achieved by fixing the intensity of the peak of the electroactive element obtained by cyclic voltammetry or by imposing the potential of the peak of the electroactive element.

The degradation of anthocyanins could be correlated with the degradation of organic acids. Indeed, Kimila et al [39] observed the influence of temperature and storage time (6 months) on the ascorbic acid in roselle extract. Organic acid losses during storage are mainly affected by anaerobic degradation caused by temperature and storage time.

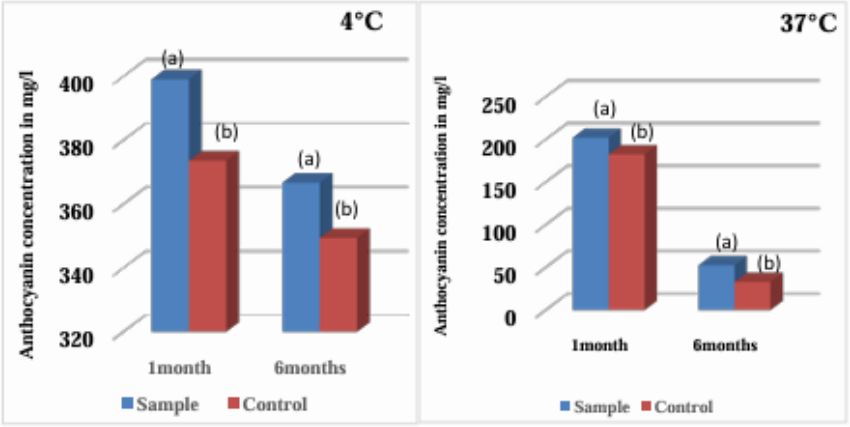

Figure 10. Monitoring of anthocyanins in electrochemically treated roselle extract (Sample) and untreated roselle extract (Control) after 1 month and 6 months of storage at 4°C and 37°C [38]

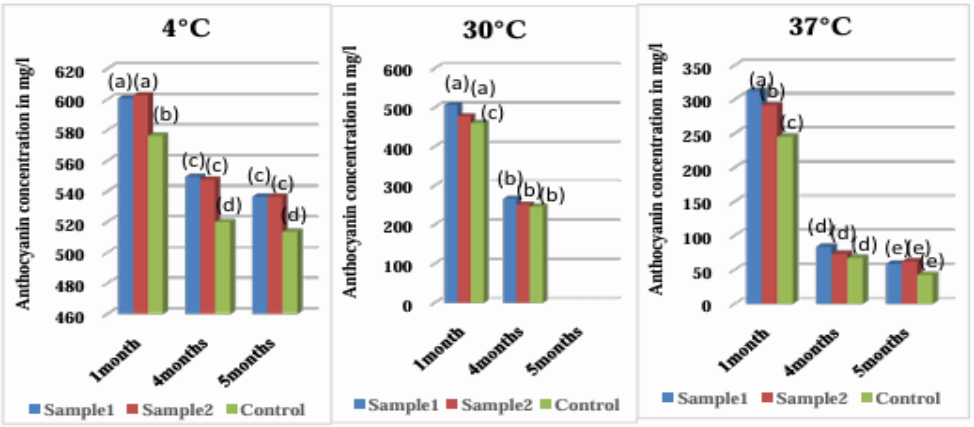

Figure 11. Monitoring of anthocyanins in the extract treated by chronoamperometry -250mV/30mn (sample 1), in the extract treated by chronopotentiometry -5mA/30mn (sample 2) and in the untreated extract (control), stored at 4°C, 30°C and 37°C for 5 months [38]

4.1.3. Modeling of Reaction Kinetics in Roselle Extract

In this section, the author [40] studied the kinetics of thermal degradation of electrically reduced extracts compared to roselle extract in which oxygen had not been reduced (control).

To do this, two samples treated respectively with a potential of -125mV for 30 minutes (sample 1) and an intensity of -5mA for 30 minutes (sample 2) were considered.

The study of the degradation kinetics of extracts stored for five months at 4°C, 30°C, and 37°C showed linear curves with regression coefficients between 0.96 and 0.99. This means that the degradation kinetics of anthocyanins follow a first-order reaction. Thus, the Arrhenius, Eyring, and Ball models are applicable.

The kinetic parameters of the three models used (Arrhenius, Eyring, and Ball) are recorded in the following tables.

Table 6. Kinetic parameters of the Eyring (enthalpies ΔH* and entropies ΔS*), Arrhenius (rate constant K∞ and activation energy Ea), and Ball (decimal reduction time Do and factor Z) models [40]

Eyring		
	ΔH^* (J.mol^{-1})	ΔS^*(J.mol^{-1}K^{-1})
Control	5,72E+04^{a}	-65,95$^{a'}$
Sample1	6,08E+04^{b}	-54,37$^{b'}$
Sample2	6,00E+04^{c}	-56,64$^{c'}$
Arrhénius		
	K∞ (s^{-1})	Ea (KJ.mol^{-1})
Control	5,92E+09^{a}	59,58$^{a'}$
Sample1	2,38E+10^{b}	63,21$^{b'}$
Sample2	1,82E+10^{c}	62,44$^{c'}$
Ball		
	Do (S)	Z (°C)
Control	1,43E+11^{a}	28,65$^{a'}$
Sample1	6,47E+11^{b}	27,03$^{b'}$
Sample2	4,48E+11^{c}	27,40$^{c'}$

The kinetic parameters showed that the electro-reduced extract is more resistant than the untreated extract. Indeed, on the one hand, the activation energy of samples 1 and 2 (63.21 and 62.44 KJ.mol^{-1}) is greater than that of the control (59.58 KJ.mol^{-1}). This means that more energy is required to activate the degradation kinetics of the electro-reduced extracts, unlike the untreated roselle extract. Similarly, the rate constants are 2.38E+10 and 1.82E+10 s^{-1} for samples 1 and 2, respectively, while that of the control is 5.92E+09 s^{-1}.

On the other hand, the Eyring model shows that electrochemical treatment is not energy-intensive, with a significant difference in enthalpies of approximately 0.28 J.mol^{-1} noted between the samples (6.08E+04 and 6.00E+04 J.mol^{-1}) and the control (5.72E+04 J.mol^{-1}).

Finally, according to Hallstrom et al., the z factor is directly related to the activation energy in Arrhenius' law. When z is between 25 and 50 °C and Ea between 60 and 110 KJ/mol, a hydrolysis reaction occurs in solution [41].

When z is between 38 and 80°C and Ea between 30 and 90 KJ/mol, the dye is destroyed, so electrochemical treatment does not destroy the dye in the hibiscus extract. Similarly, *Hibiscus sabdariffa L.* extract does not undergo non-enzymatic browning because the values of z (Ball) are not between 17 and 39°C, nor are those of Ea (Arrhénius) between 100 and 250 KJ/mol [41].

Furthermore, it is important to note that the two electrochemical treatment methods used to reduce dissolved oxygen in the extract (sample 1: -125mV/30min and sample 2: -5mA/30min) show significant differences for all models (Table 6).

It would appear that treatment by chronoamperometry (-125mV/30min) is more effective than reduction by chronopotentiometry (-5mA/30min), as shown by the results in Table 6.

5. General Conclusion

The objective of this review was to summarize the use of membrane technologies for the stabilization of *Hibiscus sabdariffa L.* extract, particularly the electrochemical approach.

In general, membrane methods are a viable alternative to thermal methods.

Scientific results have shown that the use of membranes guarantees microbial stability and preserves the nutritional qualities of the product, but does not treat dissolved oxygen, which significantly degrades anthocyanins in *Hibiscus sabdariffa L.* extract.

A new cold electrochemical treatment method using a commercial membrane reduces dissolved oxygen on a platinum/ECS electrode, thereby stabilizing anthocyanins

during storage.

During the first month of storage, a significant reduction in anthocyanin losses of 10% was achieved at 37°C with the batch reactor. This had previously been problematic for other treatments.

Monitoring anthocyanins treated with an intensity/time combination (-6 mA/30 minutes) and stored for 6 months at 4°C showed a constant reduction in anthocyanin losses of 5% from the first to the sixth month of storage, demonstrating the effectiveness of the method.

This new electrochemical approach could be a real alternative to bubbling on an industrial scale.

Membrane technologies are now essential for the circular economy. They play a crucial role in water treatment and food stabilization, promoting more sustainable practices, increasing industrial resilience, and paving the way for more efficient economic models.

References

[1] P. M. Ngom, « Essai de stabilisation de la couleur rouge de la boisson de bissap (Hibiscus sabdariffa L.) », *Thčse Dr. 3čme Cycle Univ. Cheick Anta Diop Dakar Sénégal 110p*, 2001.

[2] M. Cisse, M. Dornier, M. Sakho, C. MarDiop, M. Reynes, et O. Sock, « La production du bissap (Hibiscus *sabdariffa* L.) au Sénégal », *Fruits*, vol. 64, n° 2, p. 111-124, mars 2009.

[3] F. N. Muanda, « Identification de polyphénols, évaluation de leur activité antioxydante et étude de leurs propriétés biologiques », *Univ. Paul Verlaine-Metz*, vol. 238, 2010, Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.fr/api/v1/document/2010METZ011S.

[4] B. B. Amor, « Maîtrise de l'aptitude technologique de la matière végétale dans les opérations d'extraction de principes actifs: texturation par détente instantanée contrôlée (DIC) », PhD Thesis, Université de La Rochelle, 2008. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.hal.science/tel-00399131/.

[5] F. J. Olivas-Aguirre *et al.*, « Cyanidin-3-O-glucoside: Physical-chemistry, foodomics and health effects », *Molecules*, vol. 21, n° 9, p. 1264, 2016.

[6] F. Khater, « Identification et validation fonctionnelle de nouveaux gènes impliqués dans la voie de biosynthèse des composés phénoliques », PhD Thesis, Montpellier, SupAgro, 2011. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.fr/2011NSAM0018.

[7] K. Puro, R. Sunjukta, S. Samir, S. Ghatak, I. Shakuntala, et A. Sen, « Medicinal uses of Roselle plant (Hibiscus sabdariffa L.): a mini review », *Indian J. Hill Farming*, vol. 27, n° 1, p. 81-90, 2014.

[8] I. Da-Costa-Rocha, B. Bonnlaender, H. Sievers, I. Pischel, et M. Heinrich, « Hibiscus sabdariffa L.–A phytochemical and pharmacological review », *Food Chem.*, vol. 165, p. 424-443, 2014.

[9] B. Lin, C. Gong, H. Song, et Y. Cui, « Effects of anthocyanins on the prevention and treatment of cancer », *Br. J. Pharmacol.*, vol. 174, n° 11, p. 1226-1243, juin 2017.

[10] D. Li, P. Wang, Y. Luo, M. Zhao, et F. Chen, « Health benefits of anthocyanins and molecular mechanisms: Update from recent decade », *Crit. Rev. Food Sci. Nutr.*, vol. 57, n° 8, p. 1729-1741, mai 2017.

[11] L. G. Maciel *et al.*, « Hibiscus sabdariffa anthocyanins-rich extract: Chemical stability, in vitro antioxidant and antiproliferative activities », *Food Chem. Toxicol.*, vol. 113, p. 187-197, 2018.

[12] R. Beltrán-Debón *et al.*, « The aqueous extract of Hibiscus sabdariffa calices modulates the production of monocyte chemoattractant protein-1 in humans », *Phytomedicine*, vol. 17, n° 3-4, p. 186-191, 2010.

[13] M. Cisse, M. Dornier, M. Sakho, A. Ndiaye, M. Reynes, et O. Sock, « Le bissap (Hibiscus sabdariffa L.): composition et principales utilisations », *Fruits*, vol. 64, n° 3, p. 179-193, 2009.

[14] D. Li, P. Wang, Y. Luo, M. Zhao, et F. Chen, « Health benefits of anthocyanins and molecular mechanisms: Update from recent decade », *Crit. Rev. Food Sci. Nutr.*, vol. 57, n° 8, p. 1729-1741, mai 2017.

[15] B. Anoman Jean-Claude, T. Abdoulaye, Z. Armel Fabrice, K. Ahmont Landry Claude, C. Adama, et Z. Lessoy Yves Thierry, « Evaluation of Physicochemical, Nutritional and Antioxidant Parameters of Pulp During Post-harvest Ripening of Kent Variety Mango from Northern Côte d'Ivoire », *J. Food Nutr. Res.*, vol. 10, n° 6, p. 386-391, juin 2022.

[16] Y. S. K. Dewi, « Phytochemical characteristics and antioxidant activity of liang tea at different pH during storage », *Gorontalo Agric. Technol. J.*, p. 86-96, 2022.

[17] L.-F. Wang, D.-M. Kim, J.-D. Park, et C. Y. Lee, « various antibrowning agents and green tea extract during processing and storage », *J. Food Process. Preserv.*, vol. 27, n° 3, p. 213-225, août 2003.

[18] A. M. Sinela, « Etude des mécanismes réactionnels et des cinétiques de dégradation des anthocyanes dans un extrait d'Hibiscus sabdariffa L. », PhD Thesis, Montpellier SupAgro, 2016. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.hal.science/tel-01580166/.

[19] M. Cissé, P. Bohuon, F. Sambe, C. Kane, M. Sakho, et M. Dornier, « Aqueous extraction of anthocyanins from Hibiscus sabdariffa: Experimental kinetics and modeling », *J. Food Eng.*, vol. 109, n° 1, p. 16-21, 2012.

[20] S. Nuryanti, S. Matsjeh, C. Anwar, et T. J. Raharjo, « Isolation anthocyanin from roselle petals (Hibiscus sabdariffa L) and the effect of light on the stability », *Indones. J. Chem.*, vol. 12, n° 2, p. 167-171, 2012.

[21] D. P. Makris et J. T. Rossiter, « Hydroxyl free radical-mediated oxidative degradation of quercetin and morin: a preliminary investigation », *J. Food Compos. Anal.*, vol. 15, n° 1, p. 103-113, 2002.

[22] Z. Houli, D. Merrouche, et F. Boudjelal, « Suivi de la dynamique intrannuelle de la chlorophylle dans les eaux côtières Est de Jijel à partir d'images satellitaires », PhD Thesis, université de jijel, 2017.

[23] A. Allalgua, N. KAOUACHI, C. BOUALLEG, A. AYARI, et B. Mourad, « Caracterisation Physico-Chimique Des Eaux Du Barrage Foum El-Khanga (Region De Souk-Ahras, Algerie) », *Eur. Sci. J. ESJ*, vol. 13, n° 12, 2017, Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://www.univ-soukahras.dz/en/publication/article/1146.

[24] M. Cisse, F. Vaillant, A. Kane, O. Ndiaye, et M. Dornier, « Impact of the extraction procedure on the kinetics of anthocyanin and colour degradation of roselle extracts during storage », *J. Sci. Food Agric.*, vol. 92, n° 6, p. 1214-1221, avr. 2012.

[25] L. Pechamat, « Impacts de l'oxygène sur les évolutions chimiques et sensorielles du vin rouge », PhD Thesis, Université de Bordeaux, 2014. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.hal.science/tel-01191500/.

[26] R. Beltrán-Debón *et al.*, « The aqueous extract of Hibiscus sabdariffa calices modulates the production of monocyte chemoattractant protein-1 in humans », *Phytomedicine*, vol. 17, n° 3-4, p. 186-191, 2010.

[27] M. J. Cejudo-Bastante, M. S. Pérez-Coello, et I. Hermosín-Gutiérrez, « Effect of wine micro-oxygenation treatment and storage period on colour-related phenolics, volatile composition and sensory characteristics », *LWT-Food Sci. Technol.*, vol. 44, n° 4, p. 866-874, 2011.

[28] E. Le Deun, « Couleur des jus de pomme et des cidres: analyse structurale et impact de plusieurs paramètres physico-chimiques », PhD Thesis, Université de Rennes, 2016. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.hal.science/tel-01758930/.

[29] A. Sinela, N. Rawat, C. Mertz, N. Achir, H. Fulcrand, et M. Dornier, « Anthocyanins degradation during storage of Hibiscus sabdariffa extract and evolution of its degradation products », *Food Chem.*, vol. 214, p. 234-241, 2017.

[30] A. Bal, H. N. Shilpa, S. Debnath, et N. K. Rastogi, « Extraction of anthocyanin from Hibiscus rosa-sinensis and concentration by forward osmosis membrane process », *Innov. Food Sci. Emerg. Technol.*, vol. 96, p. 103782, 2024.

[31] M. Cisse, « Couplage de procédés membranaires pour la production d'extraits anthocyaniques: application à Hibiscus sabdariffa », PhD Thesis, Montpellier SupAgro, 2010. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://agritrop.cirad.fr/567903.

[32] A. F. Adje, Y. Houphouët-Boigny, Y. Lozano, et H. M. Biego Godi, « Couplage de technologies membranaires pour la production d'extraits stables de bissap (Hibiscus sabdariffa L.,

Malvaceae) », PAG, 2015. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https:// agritrop.cirad.fr/ 578133/ 1/LOZANO%20P.589.pdf.

[33] C. M. Paraíso *et al.*, « Intensified ultrafiltration process for fouling mitigation during concentration of bioactive compounds from hibiscus (Hibiscus sabdariffa L.) extract: Innovation by using ultrasound and 3D turbulence promoters », *Chem. Eng. Process-Process Intensif.*, vol. 195, p. 109612, 2024.

[34] M. Cisse, F. Vaillant, D. Soro, M. Reynes, et M. Dornier, « Crossflow microfiltration for the cold stabilization of roselle (Hibiscus sabdariffa L.) extract », *J. Food Eng.*, vol. 106, n° 1, p. 20-27, 2011.

[35] T. N. N. Hoang, N. P. M. Nguyen, T. A. D. Dong, et T. H. A. Le, « Anthocyanin isolation from Hibiscus sabdariffa L. flowers by extraction, macroporous D101 resin purification, and biological evaluation », *J. Agric. Food Res.*, vol. 14, p. 100848, 2023.

[36] N. Khady, K. Cheikhou, A. Nicolas, C. Mady, et D. C. Mar, « Characterisation of Electrochemical Parameters for the Stabilisation of Anthocyanins from Hibiscus sabdarrifa L », *Am. J. Food Sci. Technol.*, vol. 9, n° 4, p. 125-133, 2021.

[37] N. Le Bozec, « Réaction de réduction de l'oxygène sur les aciers inoxydables en eau de mer naturelle. Influence du biofilm sur les processus de corrosion », PhD Thesis, Université de Bretagne Occidentale, 2000. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: http://archimer.ifremer.fr/doc/00000/223/.

[38] K. Ndiaye, C. Kane, O. B. K. Cisse, N. Ayessou, et C. M. Diop, « Monitoring of anthocyanins and colour in electrochemically processed Hibiscus sabdariffa juice », *Food Nutr. Sci.*, vol. 12, n° 11, p. 1073-1087, 2021.

[39] B. Mgaya-Kilima, S. F. Remberg, B. E. Chove, et T. Wicklund, « Influence of storage temperature and time on the physicochemical and bioactive properties of roselle-fruit juice blends in plastic bottle », *Food Sci. Nutr.*, vol. 2, n° 2, p. 181-191, mars 2014.

[40] N. Khady, K. Cheikhou, N. Mouhamed, A. Nicolas, C. Mady, et D. C. Mar, « Modelling of Reaction Kinetics of Hibiscus Sabdariffa L. Juice Anthocyanins Degradation by Electrochemical Means », *J. Food Secur.*, vol. 11, n° 3, p. 85-91, nov. 2023.

[41] N. Al Fata, « Conception et exploitation d'un dispositif expérimental instrumenté pour la prévision de la dégradation de la qualité nutritionnelle et de l'inactivation microorganismes dans les fruits et légumes transformés », PhD Thesis, Université d'Avignon, 2017. Consulté le: 23 septembre 2025. [En ligne]. Disponible sur: https://theses.hal.science/tel-01704619/.

© The Author(s) 2025. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).