UNIVERSIDAD NACIONAL DE COLOMBIA
SEDE MANIZALES

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Determination of extraction kinetics of bioactive compounds from spent coffee grounds (*Coffea arábica*)

Ashley Sthefanía Caballero Galván, Daissy Lorena Restrepo Serna, Mariana Ortíz Sánchez, Carlos Ariel Cardona Alzate*

Universidad Nacional de Colombia sede Manizales, Instituto de Biotecnología y Agroindustria. Laboratorio de Equilibrios Químicos y Cinética Enzimática. Departamento de Ingeniería Química. Manizales, Colombia

The Coffee

Figure 1. Coffee plantation

- Annual production of 156.6 million bags of 60 kilograms in 2016.
- This product is responsible for the generation of large quantities of waste, as the coffee pot, grounds and mucilage

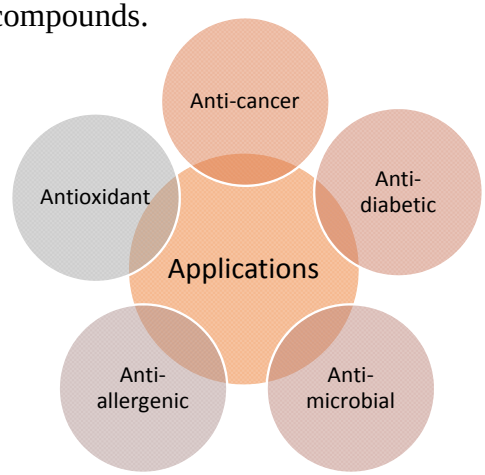
Figure 2. Spent coffee grounds

Spent coffee grounds are the major residues of the soluble coffee industry, generating 6 million tons per year and causing economic and environmental issues when this residue is discharged to the surroundings or incinerated

Several authors have reported the use of the spent coffee grounds (SCG) to obtain different added-value products and thus, increasing the sustainability of the global coffee industry. Different studies have reported large quantities of **polyphenolic components** in the SCG with a high concentration of chlorogenic acid. There is several applications of these components in the food, pharmaceutics and cosmetic industry.

Polyphenolic Compounds

- Polyphenolic compounds are part of secondary metabolites, being identified more than 8.000 compounds.



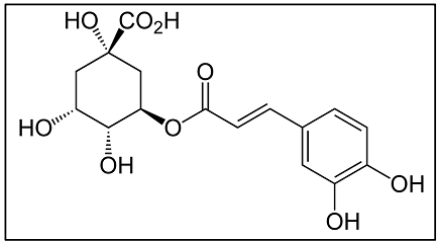
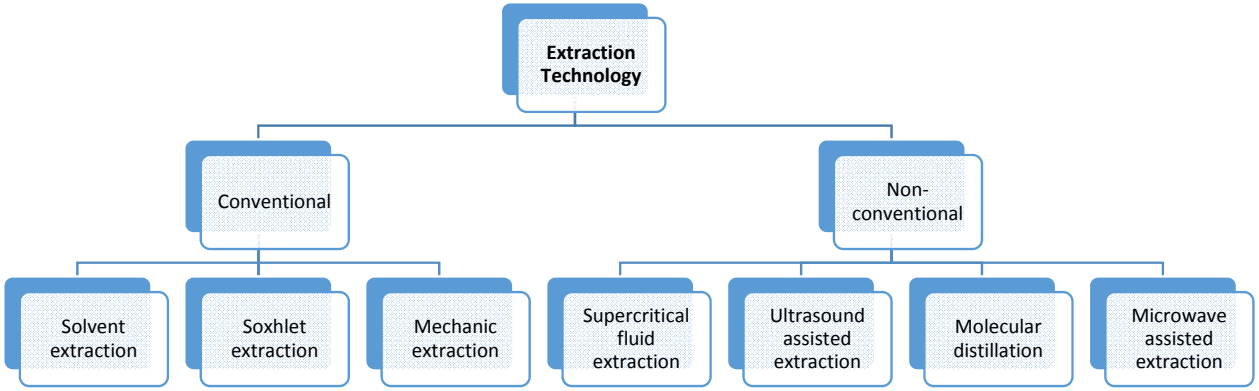


Figure 3. Chlorogenic acid structure



The determination of the extraction kinetics has high importance for the design of the extraction equipment, allowing an optimization of the time, energy and cost of the process. Additionally, the determination of the extraction kinetics provides a great knowledge about the extraction rates

Kinetic Models

Model	Equation
Gaussian model	$y= a * \exp\left(-\frac{(x-b)^2}{c}\right)$
Richards model	$y= \frac{a}{(1+\exp(b-c*x))^{\frac{1}{d}}}$
Reciprocal Quadratic model	$y= \frac{1}{a+b*x+c*x^2}$
Weibull model	$y= a - b * \exp\left(c * x^d\right)$
Exponential models	$y= a * \exp(b * x)$
Steinhart-Hart model	$y= \frac{1}{a+b*Ln(x)+c*Ln(x)^3}$

Model	Equation
Rational model	$y= \frac{a+b*x}{1+c*x+d*x^2}$
Bleasdale model	$y= (a - b * x)^{-\frac{1}{c}}$
Heat Capacity model	$y= a + b * x - \frac{c}{x^2}$
Shifted Power	$y= a * (x - b)^c$
Modified Hoerl model	$y= a * (b)^{\frac{1}{x}} * x^c$
Exponential Association 3 model	$y= a * (b - \exp(c * x)$

Collection and drying of raw material → Characterization of raw material → Polyphenolic compounds extraction → Determination of polyphenolic compounds → Obtaining extraction kinetics

Characterization of raw material

Extractives (NREL/TP-510-42619),

Cellulose (T203 os-74 ASTM 1695-77)

Holocellulose (ASTM D1104)

Lignin (T222)

Moisture

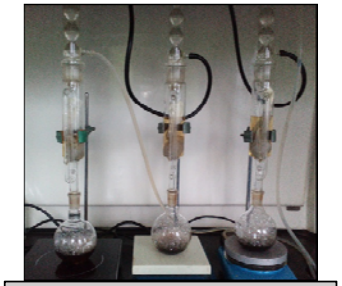
Ash (NREL/TP-510-42622),

Extraction → Vacuum filtration → Centrifugation → Analysis of extractives

Solvent Extraction

Temperature: 25 °C
Solvent/sample ratio: 20:1
Solvent: Ethanol 60% v/v
Time: 8 hours

Soxhlet Extraction



Sample weight: 10 g.
Solvent volumen: 250 mL
Solvent: Ethanol 60% v/v
Time: 5 hours

Ultrasound Assisted Extraction

Equipment: Ultrasonic processor UP50H (Hielscher Ultrasound Technology)
Ultrasonic power: 750 W
Frequency: 20 kWh
Temperature: 50 °C ± 2 °C
Solvent/sample ratio: 20:1
Solvent: Ethanol 60% v/v
Time intervals: 10, 20 30 40 50 and 60 min

Table 1. Physicochemical composition of SCG.

	This work*	Reference [6]	Reference[2]
Extractives	25.64 ± 0.27	ND	---
Holocellulose	50.32 ± 2.03	---	---
Cellulose	21.48 ± 0.06	8.6 (Glucose)	12.40 ± 0.70 (Glucose)
Hemicellulose	28.84 ± 2.09	36.7	39.10 ± 1.90
Lignin	19.83 ± 0.76	ND	23.90 ± 1.70
Ash	0.94 ± 0.05	1.6	30. ± 0.10
***All the percentages are expressed by weight.**			

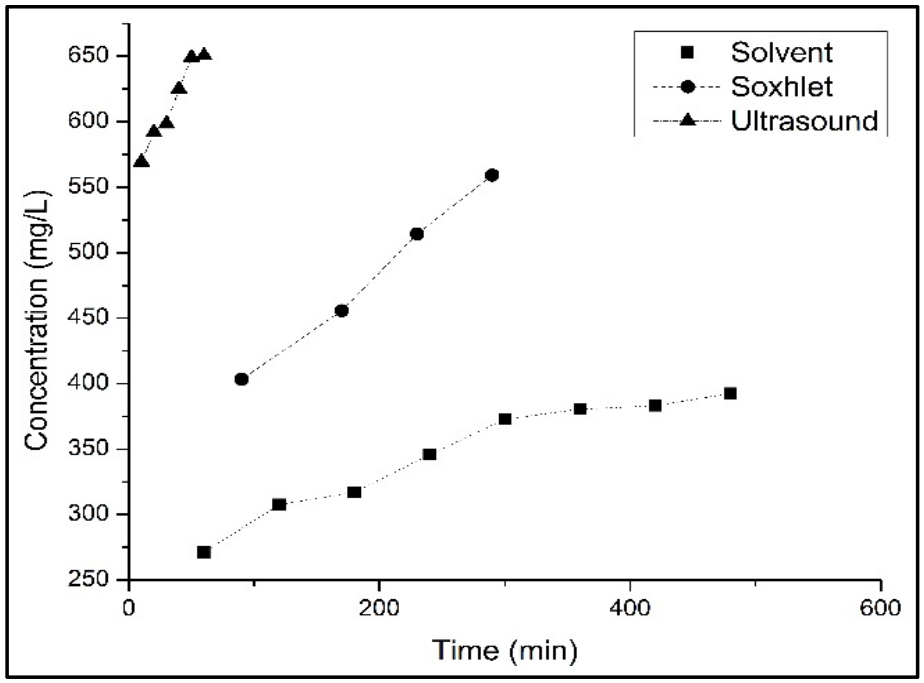
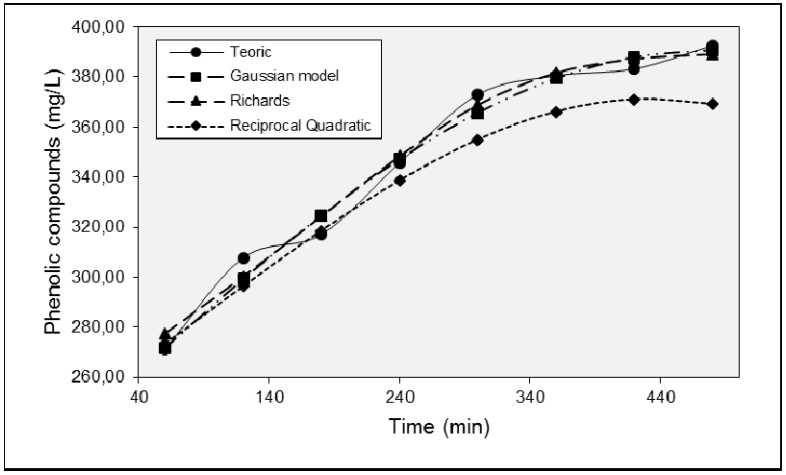


Figure 1. Phenolics compounds concentration vs time.

Extraction kinetics of phenolic compounds (Folin-Ciocalteu)



Solvent extraction (TPC total phenolic compounds)

- Gaussian model

$$TPC = 390.74 * \exp\left(-\frac{(x-478.66)^2}{2*494.07^2}\right) \quad Eq.1$$

R^2=0.984

- Richards model

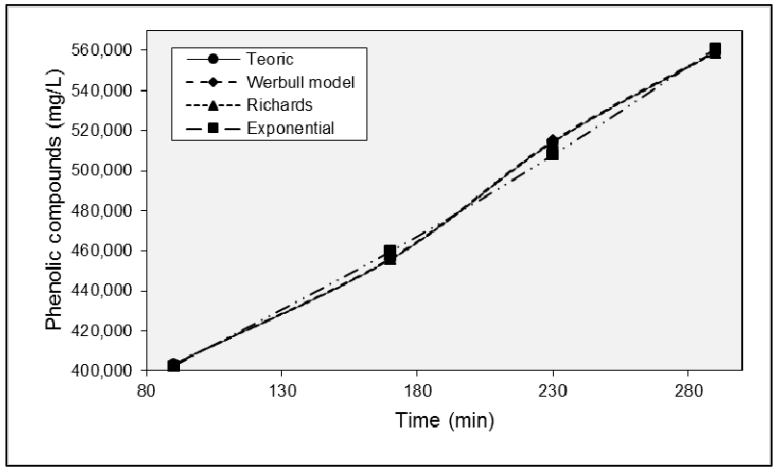
$$TPC = \frac{390.05}{(1+\exp(6.56-0.02*x))^{\frac{1}{15.22}}} \quad Eq.2$$

R^2=0.986

- Reciprocal Quadratic model

$$TPC = \frac{1}{4.00*10^{-3}+6.30*10^{-6}*x+6.92*10^{-9}*x^2} \quad Eq.3$$

R^2=0.984



Soxhlet Extraction

- Weibull model

$$TPC = 582.55 - 189.54 * \exp(-5.20*10^{-8}*x^{3.09}) \quad Eq.4$$

R^2=0.999

- Richards model

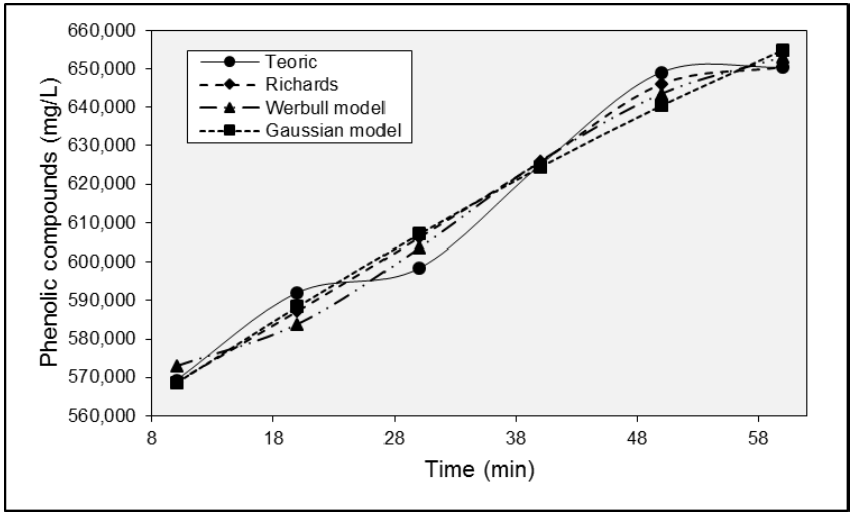
$$TPC = \frac{559.31}{(1+\exp(83.89-0.29*x))^{\frac{1}{171.23}}} \quad Eq.5$$

R^2=0.997

- Exponential models

$$TPC = 346.80 * \exp(1.66*10^{-3}*x) \quad Eq.6$$

R^2=0.995



Ultrasound assisted extraction

- Richards model

$$TPC = \frac{650.45}{(1 + \exp(292.44 - 5.61 * x))^{\frac{1}{1{,}761.17}}} \qquad Eq.7$$

R^2=0.982

- Weibull model

$$TPC = 657.59 - 86.90 * \exp(-6.42 * 10^{-5} * x^{2.62}) \qquad Eq.8$$

R^2=0.973

- Gaussian model

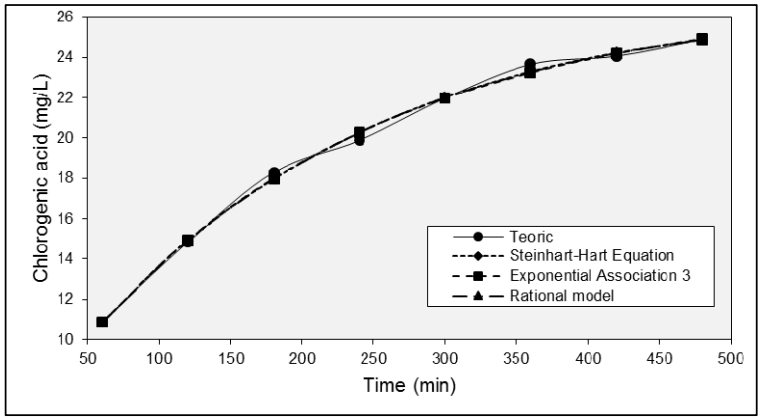
$$TPC = 702.73 * \exp\left(-\frac{(x - 128.40)^2}{2 * 182.02^2}\right) \qquad Eq.9$$

R^2=0.966

Technology	Phenolic compound (mg/L)
Solvent extraction	392.50
Soxhlet extraction	559.09
UAE	**650.453**

Extraction kinetics of chlorogenic acid

Solvent Extraction (CAC chlorogenic acid content)



- Steinhart-Hart model

$$CAC = \frac{1}{0.33 - 6.80 * 10^{-2} * Ln(x) + 5.38 * 10^{-4} * Ln(x)^3} \qquad Eq.10$$

R^2=0.997

- Exponential Association 3 model

$$CAC = 21.65 * (1.25 - \exp(-4.86 * 10^{-3} * x) \qquad Eq.11$$

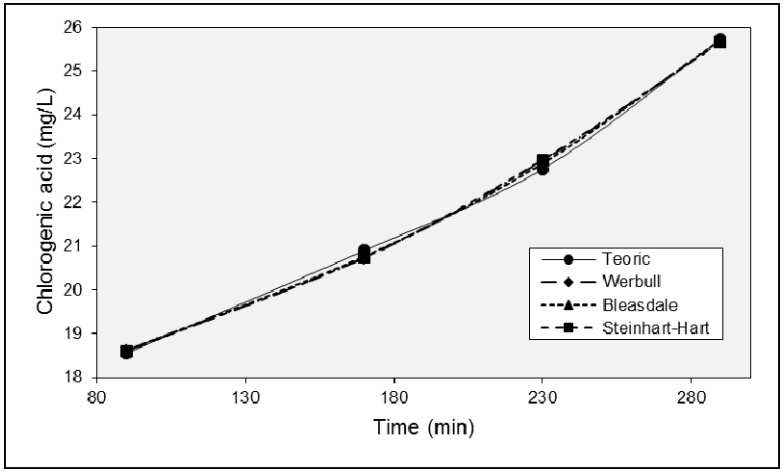
R^2=0.997

- Rational model

$$CAC = \frac{5.61 + 0.11 * x}{1 + 2.13 * 10^{-3} * x + 1.59 * 10^{-6} * x^2} \qquad Eq.12$$

R^2=0.997

Soxhlet Extraction



- Weibull model

$$CAC = 243.85 - 226.74 * \exp(-1.33 * 10^{-6} * x^{1.80}) \qquad Eq.13$$

R^2=0.997

- Bleasdale model

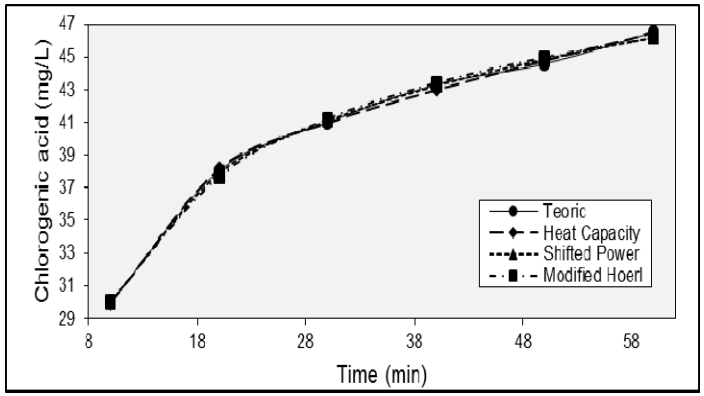
$$CAC = (1.13 * 10^{-2} - 1.97 * 10^{-5} * x)^{-\frac{1}{1.60}} \qquad Eq.14$$

R^2=0.998

- Steinhart-Hart model

$$CAC = \frac{1}{-1.37 * 10^{-2} + 2.54 * 10^{-2} * Ln(x) - 4.96 * 10^{-4} * Ln(x)^3} \qquad Eq.15$$

R^2=0.997



Ultrasound assisted extraction

- Heat Capacity model

$$CAC = 37.15 + 0.16 * x - \frac{882.04}{x^2} \qquad Eq.16$$

R^2=0.999

- Shifted Power

$$CAC = 26.83 * (x - 7.76)^{0.14} \qquad Eq.17$$

R^2=0.998

- Modified Hoerl model

$$CAC = 34.57 * (3.54 * 10^{-2})^{\frac{1}{x}} * x^{8.42*10^{-2}} \qquad Eq.18$$

R^2=0.997

Table 2. Chlorogenic acid content through different technology from coffee grounds..

Technology	Chlorogenic acid (mg/L)
Solvent extraction	24.87
Soxhlet extraction	25.22
UAE	**46.53**

The ultrasound assisted extraction method, with ethanol 60%v/v as dissolvent, proved to be an effective alternative method for the extraction of polyphenolic compounds

Spent coffee grounds presented a high concentration of chlorogenic acid making these coffee residues an attractive raw material for the food and pharmaceutical industry.

The kinetics obtained are very useful for future equipment design and overall process analysis

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Thanks for your attention

Ashley Sthefanía Caballero Galván, Daissy Lorena Restrepo Serna, Mariana Ortíz Sánchez, **Carlos Ariel Cardona Alzate***

ccardonaal@unal.edu.co

Universidad Nacional de Colombia sede Manizales, Instituto de Biotecnología y Agroindustria. Laboratorio de Equilibrios Químicos y Cinética Enzimática. Departamento de Ingeniería Química. Manizales, Colombia