Matter Transfer in Liquid-solid Extraction of Democratic Republic of Congo Gourd Seeds Oils by *"Kunyima Method"*

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Authors' contributions

This work was carried out in collaboration between all authors. Author ABK designed and supervised the study, wrote the protocol, and wrote the first draft of the manuscript. Author HMK is the experimentalist of the results of this paper. He is the Master student in the laboratory (LACOPA and Physical Cardiochemistry). Author SNL managed, discussed the results of study, performed the statistical analysis and managed the literature searching. Author PKK managed the proof reading and correction of manuscript. All authors read and approved the final manuscript.

Original Research Article

ABSTRACT

Background: The Democratic Republic of Congo has a very rich and diversified vegetable patrimony with known therapeutic properties needing only appropriate technology to deal with the extraction process of oils or active principles. This vegetable patrimony is capable to cure incurable diseases for modern medicine such as VIH, some cancers, diabetes, and so forth. So the interest, at

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first sight, has been brought on oils extraction of gourd seeds. These are widespread in all provinces of the country and are reputed to have excellent cure properties against prostate, world true calamity for men millions life. They prevent also diabetes, cardiac fits, cerebral vascular accident, liver complaints, menopause symptoms and so forth.

Aim and Objective: In this paper, the kinetic study of oil transfer from liquid- solid extraction has been undertaken in view of the phenomenon uptake to make possible home technology of reactors sizing, nowadays absent in underdeveloped countries.

Methodology: The experiments have been performed in dilute medium (petroleum ether) for a better understanding of the quantitative comparison. The liquid- solid extraction concerned here is the soxhlet extraction of oil from Gourd seeds. The figures have been plotted by means of Origin 8 program.

Results: Experimental results hereby discussed seem satisfactory and describe perfectly the proposed model. The proposed calculations have taken into account the combination of solubilisation and diffusion of matter molecules. The kinetic foundation of oil extraction and the sizing of extraction reactors are set together in KUNYIMA Method.

The extraction kinetic study of gourd seeds oil has been successfully performed in dilute solution where the linearity at more than 98% at exactly 98.8% between $\log \frac{1}{m_o - m}$ and time expressed in hours was pointed out. The model was valid and the kinetic constant calculated.

Conclusion: KUNYIMA Method consists of the best uptake of the phenomenon, in expressing it in a suitable mathematical model to determine its velocity through its kinetic constant before sizing the experimentation reactor. The reactor volume depends on both the sizing factor and the desired volumic debit. This last parameter depends strongly on kinetic constant and conversion degree. The interest is emphasised of course for the moment on medicinal plants, but the method can cover any other similar field.

1. INTRODUCTION

Prior to this work, it has been reported several time the benefits of Gourd seeds. They contain magnesium necessary for healthy heart and participating to vital physiological functions namely the manufacture (making) of TPA, the synthesis of RNA and DNA, the heart beat, formation of bones and teeth, the relaxation of blood vessels and a good intestinal transit. Magnesium promotes a healthy arterial pressure and helps to prevent cardiac Stoppages, Cardiac fits and cerebral vascular accidents [1,2].

They also contain zinc acting as a stimulating agent of immunity, very prominent for the growth and cellular division, sleep, humour, smell, taste, skin, eyes health, the insulin regulation (decreasing considerably oxidative stress) and the male sexual function. They can improve sperm quality and fertility. Zinc is very needful for prostate health. Gourd seeds have omega-3 fatty acids of vegetable (plant) origin (Alpha Linolenic Acid: ALA) very necessary for the body. They are rich in natural phytoestrogen capable to entail an increase of HDL (good cholesterol) and to monitor the menopause symptoms (arterial pressure, sudden flush, headache, articular pains, etc.). They are rich in antioxidants and fibres (good for heart and liver health). They contain tryptophan for restoring sleep, convertible by the organism in serotonin first of all and afterwards in melatonin which is a sleep hormone. Gourd seeds oils cause anti-inflammatory effects [1,3].

They can prevent and treat urinary troubles and they act against intestinal parasites. They contain also elements such as potassium, manganese, selenium, chromium, molybdenum, tocopherols and phytosterols. Modern medicine with its advantages and drawbacks is not attainable to the underdeveloped countries populations where appropriate technology (home technology) is cruelly absent for the need of industrial production because of their increasing impoverishment and because of the lack of imagination [4,5,6,7].

Hence it seems consistent to engage manfully Democratic Republic of Congo on phytotherapy medicine, very cheap and accessible.

The country has a very rich and diversified vegetable patrimony with known therapeutic

properties needing only appropriate technology to deal with the extraction process of oils or active principles. This vegetable patrimony is capable to cure incurable diseases for modern medicine such as VIH, some cancers, diabetes, and so forth. So the interest, at first sight, has been brought on oils extraction of gourd seeds for evident above mentioned reasons [6,7,8].

2. MATERIALS AND METHODS

2.1 Materials

Drying oven, Balance (mark OHAUS), watch glass, desiccator, heating skull cap, spade, beaker, cellulose cartridge (33 X 205 mm), thermometer, gel of silicone, aluminium paper, thermostat, rotary evaporator, chronometer, crushing machine, burettes, distillate water and petroleum ether have been used. Origin 8 program has been used.

Gourd seeds with the following characteristics have been used in this research [9,10,11]:

Family: *Cucurbitaceaea*
Order: Violales
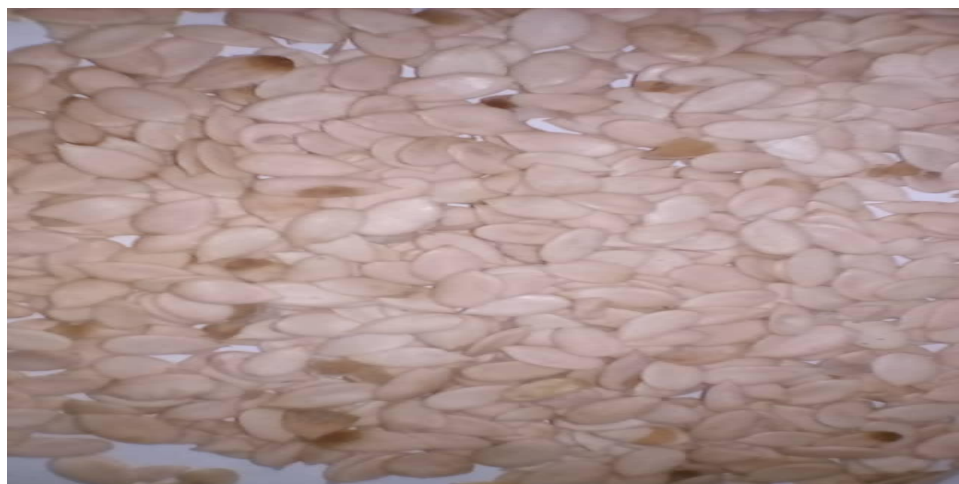Class: Magnoliopsida
Gender: *Cucurbita*
Species: *pepo*

Fig. 1a. Gourd seeds of *Cucurbita pepo*, DRC

Fig. 1b. Soxhlet extraction setting used

2.2 Methods

2.2.1 Kinetic foundation

It is needful to find a mathematical model to describe the extraction of this oils in a suitable solvent. For an oil of a given nature and species, in a given solvent and for a given space, its extraction is an only time-dependent phenomenon, and it can be linear [12,13,14,15].

Therefore, it can be expressed by instantaneous variation of quantity i.e the difference between comers and goers massic debits. Quatity variation = inlet massic debit – exit massic debit [16,17]:

$$\frac{dx_i}{dt} = \sum_{\substack{j=1 \\ j \neq i}}^{n} k_{ji} x_j - \sum_{\substack{j=1 \\ j \neq i}}^{n} k_{ij} x_i \qquad (1)$$

where i and j are the compartments (i= solvent; j= solid state).

For the system in the study there is no entrance of oils, there is only exit of oil by extraction.

Logically taking into account the mass balance it can be written that the above – mentioned equation (1) becomes:

$$\frac{dx}{dt} = -kx \qquad (2)$$

$$m = m_0 - x$$

where m is the mass of oils at a certain time (t), m_o is the total extractible mass of oil in the sample at the beginning, considered as constant, x is the extracted mass of oils.

$$x = m_0 - m$$

Equation (2) can be written

$$\frac{dx}{dt} = \frac{d(m_o - m)}{dt} = -\frac{dm}{dt} = k(m_0 - x) \qquad (3)$$

$$\int \frac{dx}{m_0 - x} = \int k\, dt$$

$$-\ln(m_0 - x) = kt + \ln A$$

at t = 0 → x = 0

$$-\ln m_0 = \ln A$$

$$-\ln(m_0 - x) = kt - \ln m_0$$

$$\log\left(\frac{m_0}{m_0 - x}\right) = \frac{k}{2,3} t \qquad (4)$$

or

$$\log\left(\frac{1}{m_0 - x}\right) = \frac{k}{2,3} t + \log \frac{1}{m_0} \qquad (5)$$

The above reasoning leads to write

$$x = m_x = m_0(1 - e^{-kt}) \qquad (6)$$

Equation (6) can serve to verify the validity of the experiments. The kinetic study will help to size the extraction reactors and to establish the home technology or to foresee the solvent efficiency (solvent effect).

2.2.2 The sizing of extraction reactors

It has been demonstrated under isothermal conditions at constant volume (constant density) that for discontinuous stirrer vat reactors, on which interest is brought right now, the dwelling time (t) of a substance in the reactor is given by the ratio between reactor volume (V_r) and volumic debit (Q) [13,14,16].

$$t = \frac{V_r}{Q} = \frac{C_0 - C}{\Gamma} = \frac{C_0 - C}{kc} \qquad (7)$$

where C_o is the initial concentration $= \frac{Masse}{Volume}$

$$C_o = \frac{F}{Q} = \frac{massic\ debit}{volumic\ debit} \qquad (8)$$

with C= current concentration

Γ is the extraction rate in this study or else the velocity of the happening event.

$$\Gamma = kc = kC_0(1 - \gamma) \qquad (9)$$

k is the kinetic constant
γ is the conversion degree (percentage of conversion).

$$\gamma = \frac{C_0 - C}{C_0} \qquad (10)$$

$$\gamma C_0 = C_0 - C \qquad (11)$$

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(9) and (11) in (7)

$$\frac{V_r}{Q} = \frac{\gamma C_0}{kC_0(1-\gamma)} = \frac{\gamma}{k(1-\gamma)} \qquad (12)$$

In the case of oil extraction studied, the same reasoning can be done in term of mass

$$\frac{V_r}{Q} = \frac{m_0 - m}{\Gamma} = \frac{m_0 \gamma}{km_0(1-\gamma)} = \frac{\gamma}{k(1-\gamma)} \qquad (13)$$

where Γ is the extraction velocity $= km = km_0(1-\gamma)$

m_o is the initial extractible mass of oil, k is the kinetic constant expressing the easiness with which the solvent is dealing.

$$\gamma = \frac{m_0 - m}{m_0}$$

Clearly, it can be seen the relation between V_r and Q

$$V_r = \frac{\gamma}{k(1-\gamma)} Q \qquad (14)$$

For a given γ and k the relation (14) can be written $V_r = AQ$

where *A* has been called *sizing constant* or *sizing factor* as it has been decided in the laboratory.

Equation (14) shows that at a given kinetic constant, for a needed conversion, the reactor volume depends on desired volumic debit.

It is the relation of sizing showing that the knowledge of kinetic constant and the conversion degree is very important and those two parameters must be determined before sizing preoccupation.

It becomes necessary to show there is a dependence between the conversion degree and the dwelling time and therefore the reactor size [18,19,20].

$$t = \frac{V_r}{Q} = \frac{C_0 - C}{\Gamma} = \frac{C_0 - C}{kc}$$
$$tkc = C_0 - C$$

$$C_0 = tkc + C = C(1 + kt) \qquad (15)$$

$$C = \frac{C_0}{1+kt} = \frac{C_0}{1+k\left(\dfrac{Vr}{Q}\right)} \qquad (16)$$

$$\gamma = \frac{C_0 - C}{C_0} = 1 - \frac{C}{C_0} = 1 - \frac{C_0}{C_0\left[1+k\left(\dfrac{Vr}{Q}\right)\right]}$$

$$\gamma = 1 - \frac{1}{1+k\left(\dfrac{Vr}{Q}\right)} = \frac{1+k\left(\dfrac{Vr}{Q}\right) - 1}{1+k\left(\dfrac{Vr}{Q}\right)}$$

$$\gamma = \frac{k\left(\dfrac{Vr}{Q}\right)}{1+k\left(\dfrac{Vr}{Q}\right)} \qquad (17)$$

It is clear that the conversion degree increases when $k\left(\dfrac{Vr}{Q}\right)$ increasing as it can be seen in Fig. 1c.

Also the influence of kinetic constant (*k*) on conversion degree (γ) can be signalized.

In the case of the tubular reactor at constant volume [16,18,19,20,21].

$$\gamma = \frac{m_0 - m}{m_0}$$

$$t = \frac{V_r}{Q} = m_o \int_0^\gamma \frac{d\gamma}{\Gamma} = m_0 \int_0^\gamma \frac{d\gamma}{km_0(1-\gamma)} \qquad (18)$$

$$t = \frac{V_r}{Q} = -\frac{1}{k}\left[\ln(1-\gamma)\right]_0^\gamma = -\frac{1}{k}\ln(1-\gamma)$$

$$V_r = -\frac{1}{k}\ln(1-\gamma)Q \qquad (19)$$

The relations (14) and (19) show the V_r depends on the reactor nature.

2.2.3 Statistical errors calculations

In Table 2 $m_{x_{th}}$ has been calculated according to the relation:

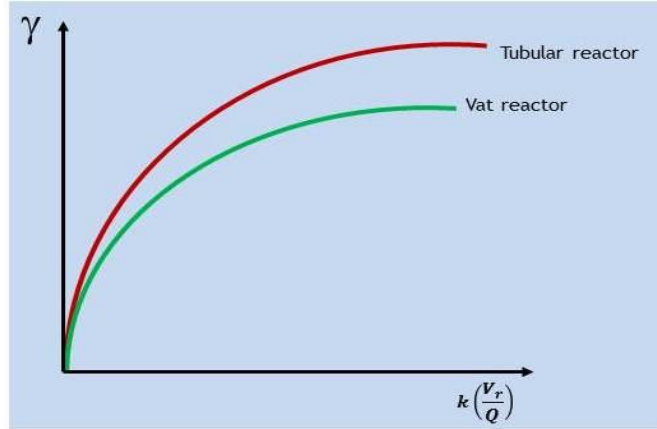$$m_{xs} = m_{x_{th}} = m_{o_{th}}(1 - e^{-kt}) \qquad (20)$$

Fig. 1c. Conversion degree versus $k\left(\dfrac{Vr}{Q}\right)$

The value of kinetic constant with its error has been given by the origin 8 program.

The value of $m_{o\,th}$ is mean value of two calculations according to the error on kinetic constant given by using the program.

For example, the intercept is – 0.7724±0.0689
That means $-\log m_{o} = -0.7724 \pm 0.0689$

$$\log m_{o} = 0.7724 \pm 0.0689$$

$$\overline{m}_{os} = \overline{m}_{o_{th}} = 10^{(0.7724 \pm 0.0689)}$$

$$m_{o_{1s}} = m_{o_{1th}} = 10^{(0.7724 + 0.0689)} = 10^{(0.8413)} = 6.9390$$

$$m_{o_{2s}} = m_{o_{2th}} = 10^{(0.7724 - 0.0689)} = 10^{(0.7035)} = 5.0524$$

$$\overline{m}_{os} = \overline{m}_{o_{th}} = \frac{m_{o_{1s}} + m_{o_{2s}}}{2} = 5.9957 \pm 0.9433 \qquad (21)$$

Mean error has been used as precision on $\overline{m}_{o\,th}$.

The error on $m_{x\,th}$ (Δm_{th}) has been calculated according to the relation:

$$\Delta m_{x_{th}} = \left(\frac{\Delta m_{o_{th}}}{m_{o_{th}}} + \frac{\Delta e^{-kt}}{1 - e^{-kt}} \right) m_{x_{th}} \qquad (22)$$

Relation (22) comes from equation (6) using the logarithmic method.

The error on exponential has been calculated as follows

$$y = e^{-kt} \; ; \quad \ln y = -kt$$
$$\frac{dy}{y} = -kdt - tdk \; ; \quad \Delta y = |t\Delta k| y = t\Delta k e^{-kt}$$

Finally the error on $m_{x_{th}}$ has been calculated by the following relation considering that there is no error on time:

$$\Delta m_{x_{th}} = \left(\frac{\Delta m_{o_{th}}}{m_{o_{th}}} + \frac{t\Delta k e^{-kt}}{1 - e^{-kt}} \right) m_{x_{th}} \quad (23)$$

2.2.4 Oil extraction protocol

Gourd seeds have been peeled, washed with distillate water, afterwards dried at 50°C for six days in the drying oven (mark memmert). These seeds were crushed and the obtained powder was preserved in a desiccator. Ten grammes of this powder were introduced in cellulose porous cartridge of 33 X 205 mm and put in soxhlet extractor.

In a three necked balloon- flask fitted of a thermometer, 400 mL of petroleum ether (40 - 60°C, ρ = 0.65 kg/L) solvent were used for the extraction.

The fitting out of soxhlet was done on heating skull cap (mark thermo scientific) in fixing the temperature at 56°C. To maintain constant temperature during the experiment, the heating skull cap was covered of aluminium as a heat insulator. The ambient temperature has been kept at 22 - 23°C [22,23,24].

After a given extraction time, the cartridge was dried in a drying oven at 50°C for 24 hours in order to get rid of traces of solvent.

The solvent in the oil-solvent mixture was recovered using a rotary evaporator at 60 °C for 24 hours. After this, the balloon flask with oil has been cooled in a desiccator and weighed. The difference between the balloon flask containing oil and the empty one determines the extracted oil mass at a t time in gramme.

3. RESULTS AND DISCUSSION

Prior to this work a lot of publications have been rendered where linear regressions between independent variables and dependent variables have been observed.

It has been considered as valid the linearity at 99%, 98%, 95%, 90%, 88% and even at 80% [25,26,27,28].

In this paper, the extraction kinetic study of gourd seeds oil has been successfully performed in dilute solution where the linearity at more than 98% at exactly 98.8% between $\log \frac{1}{m_o - m}$ and time expressed in hours was pointed out. The model was valid and the kinetic constant calculated. Table 1 shows the measured and calculated parameters as fast as the extraction advanced.

The meaning of the parameters in this table has been given in methods part.

Each value of m_x (m_o also) in this Table 1 is a mean value of three measurements. When $log \frac{1}{m_o - m_x}$ is plotted as a function of time (in hours) a straight line is found ($\approx$99%) whose the slope gives the kinetic constant k = 1.6411 $\pm$ 0.0712 h^{-1} as it can be seen in the Fig. 2a. In the same figure, the intercept is -0.7724 $\pm$ 0.0689 (the value used as an example in statistical errors calculations part).

Table 1. Mean values of m_x, m_o and their corresponding calculations (experimental values)

Time (h)	m_x (gr)	m_0 (gr)	$m_0 - m_x$	$\dfrac{1}{m_0 - m_x}$	$\log \dfrac{1}{m_0 - m_x}$	$\log \dfrac{1}{m_0}$
00h00	0.0000± 0.0000	5.3118± 0.4193	5.3118	0.1883	-0.7251	-0.7252
01h00	4.0485± 0.8790	5.3118± 0.4193	1.2633	0.7916	-0.1015	-0.7252
01h30	4.6640± 0.5862	5.3118± 0.4193	0.6478	1.5432	0.1884	-0.7252
02h00	5.1423± 0.5888	5.3118± 0.4193	0.1695	5.8997	0.7708	-0.7252
02h30	5.2212± 0.3131	5.3118± 0.4193	0.0906	11.0375	1.0429	-0.7252
03h00	5.2587± 0.3562	5.3118± 0.4193	0.0531	18.8323	1.2749	-0.7252
03h30	5.2950± 0.4075	5.3118± 0.4193	0.0168	59.5238	1.7747	-0.7252
04h00	5.3118± 0.4193	5.3118± 0.4193	0.0000	-	-	-0.7252

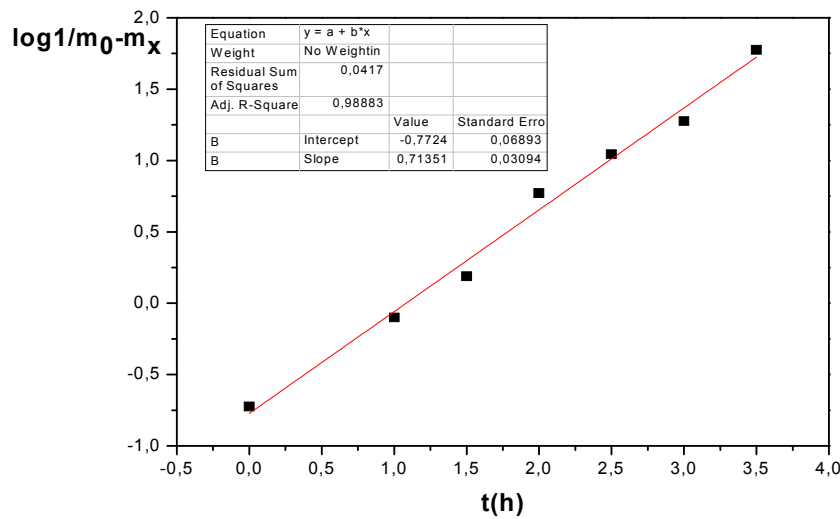| Equation	y = a + b*x	
Weight	No Weightin	
Residual Sum of Squares	0,0417	
Adj. R-Square	0,98883	
	Value	Standard Erro
B Intercept	-0,7724	0,06893
B Slope	0,71351	0,03094

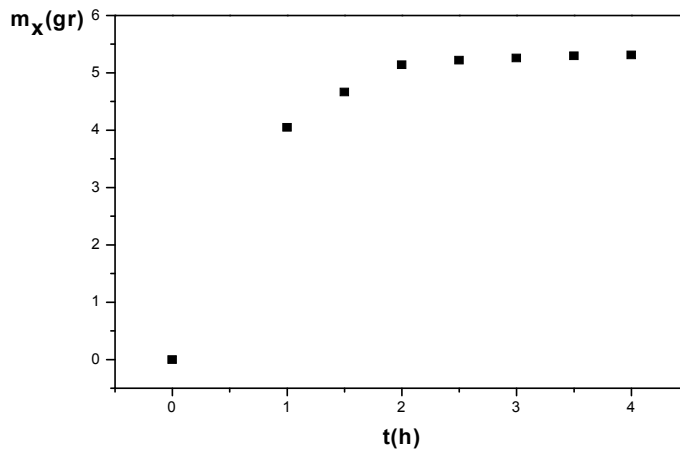**Fig. 2a.** $\log \left(\dfrac{1}{m_o - m_x} \right)$ **versus time (h)**

Fig. 2b. m_x **versus time (h)**

Origin 8 program gives itself the error.

The value of experimental m_o has been given by Table 1 and has been found the same in the plot of experimental m_x versus time as it is shown in the Fig. 2b.

In the Table 2 it can be observed the difference between experimental mass (m_e) and statistical mass (m_s). If the statistical kinetic constant is known, the statistical m_x can be calculated, in this paper, the kinetic constant from Fig.2a has been used to calculate statistical masses (m_s)

using also statistical m_o or $\overline{m}_{os} = \overline{m}_{o_{th}}$ (see statistical errors calculations part). Clearly note that the mean experimental values of mass allow to draw by origin 8 program a straight line and give both the kinetic constant and the intercept. Those last parameters are used to calculate statistical $m_s = m_{xs} = m_{x_{th}}$ and

$$m_o = m_e = \overline{m}_{os} = \overline{m}_{o_{th}} .$$

Table 2. Comparison between statistical mass (m_s) and experimental mass (m_e) as a function of time expressed in hours

Time (h)	m_s (gr)	m_e (gr)	Δm (gr)
00h00	0.0000±0.0000	0.0000±0.0000	0
01h00	4.8339±0.8432	4.0485±0.8790	0.7854
01h30	5.4843±0.9174	4.6640±0.5862	0.8203
02h00	5.7706±0.9399	5.1423±0.5888	0.6283
02h30	5.8966±0.9453	5.2212±0.3131	0.6754
03h00	5.9521±0.9458	5.2587±0.3562	0.6934
03h30	5.9765±0.9451	5.2950±0.4075	0.6815
04h00	5.9872±0.9444	5.3118±0.4193	0.6754
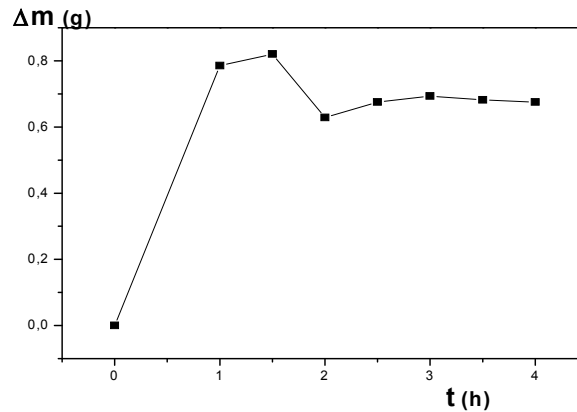
Fig. 3. Solvent effect (Δm) versus time

Smaller is the difference (Δm) better is the solvent, greater is the difference less extractant is the solvent; when the difference is zero the solvent is ideal.

The difference between statistical mass (m_s) and experimental mass (m_e) can be used as an indication to diagnose the solvent effect if it is significant. On this foundation, a quantitative comparison of extractant solvents can be made. Unfortunately, in our case this difference is not significant.

In Table 2 indeed it can be seen that the values of the difference $\Delta m = m_s - m_e$ increase generally with the increasing time until 1.5 hours and decrease after 2 hours, increase again until 3 hours and ultimately slightly decrease until 4 hours. Those differences are of the order of errors magnitude. However, if those differences are significant, the solubilisation being a statistical phenomenon, it can be observed in Fig. 3 first a random variation zone and afterwards a relative stability zone (transfer zone). In any case, it can be observed in Fig. 3 in spite of the weak difference between m_s and m_e two zones.

Observing indeed these two zones it can be seen two distinguishable events. The first corresponding to the increase of the solvent effect can be ascribable likely to the solubilisation statistical phenomenon (random variation zone) and the second showing a relative stability zone is probably due to the transfer phenomenon (diffusion-saturation). Note that this Fig. 3 has been made without taking into account errors.

Also, the value of the conversion degree is the mean value of three measurements ($\gamma = 0.5308 \pm 0.0420$). This value has been calculated according to this relation in the entitled study:

$$\gamma = \frac{m}{M} x100 \qquad (24)$$

Where

m is the oil mass obtained and M is the sample mass.

Now it is possible to calculate the reactor volume for a desired volumic debit as it is shown Table 3.
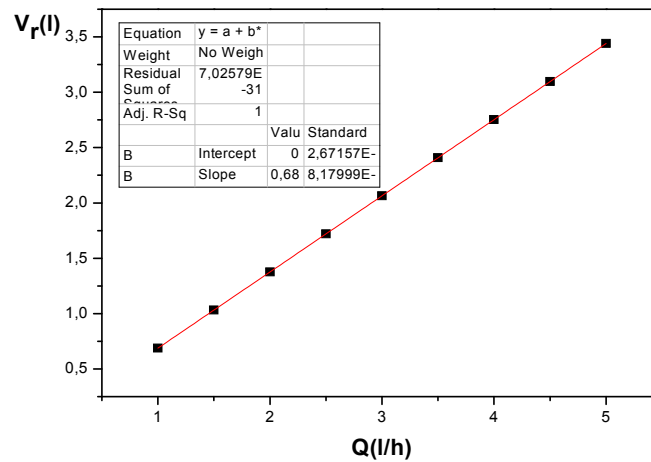
Fig. 4. V_r versus Q

Table 3. Reactor volume as a function of volumic debit

Q (l/h)	V_r (l)
0	0.0000 ± 0.0000
1	0.6880 ± 0.1458
1.5	1.0320 ± 0.2187
2	1.3760 ± 0.2917
2.5	1.7200 ± 0.3646
3	2.0640 ± 0.4375
3.5	2.4080 ± 0.5105
4	2.7520 ± 0.5834
4.5	3.0960 ± 0.6563
5	3.4400 ± 0.7293

Logarithmic method has been used to assess the precision on reactor volume (V_r)

The Fig. 4 shows linear behaviour between V_r and Q.

4. CONCLUSION

KUNYIMA Method consists in the best uptake of the phenomenon, in expressing it in a suitable mathematical model in order to determine its velocity through its kinetic constant before sizing the experimentation reactor [16,26]. It is important to work in dilute medium.

Gourd seeds oils extraction has been considered in this paper as only time dependent phenomenon (see the relation 1). Nature itself encompasses space-time dependent phenomena frequently described by the following equation:

$$\frac{\partial y}{\partial t} = a \frac{\partial^2 y}{\partial x^2} + b \frac{\partial y}{\partial x} + c \qquad (25)$$

There are also space-time independent phenomena issuing in integral equations in physiology. For example a rigid tube of *l* length and of *R* radius conveying blood loaded of dye. The dye is injected and blended in $x = 0$. The problem is to find a relation between the entrance concentration (in $x = 0$) and the exit concentration (in $x = l$). Contrarily to what is happening for gas exchange or kidney, the tube is tight. According to a biomedical point of view, this tube of small diameter (of the order of magnitude of millimeter) is called catheter and is used to measure ventricle or pulmonary volumes [16,29,30,31,32].

The velocity in this tube is supposed to be constant and space-time independent, but it can vary in a section. It is also supposed independent of r. If $C(x,t)$ is the dye concentration in the tube at point x, at t instant and if the mass balance is made, it has been established the equation of the dye evolution in the tube:

$$\frac{\partial C}{\partial t} = -v \frac{\partial C}{\partial x} \qquad (26)$$

The solution of this equation shows that

$$C(l,t) = C\left(0, t - \frac{l}{v}\right) \qquad (27)$$

The velocity is not constant

$$V(r) = \frac{2Q}{\pi R^2}\left(l - \frac{r^2}{R^2}\right) \tag{28}$$

where Q is the blood debit and this formula often used is valid if the flow is tangle [16,21,32].

Examples can be multiplied and mathematical equations should be adapted to the model analysis.

For the study in title, the reactor volume depends on both the sizing factor and the desired volumic debit. This last parameter depends strongly on kinetic constant and conversion degree.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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