

## PRODUCTION OF A HIGHLY CONCENTRATED GOLD SOLUTION FROM AQUA REGIA GOLD LEACHATE USING SUGARCANE BAGASSE NANOPARTICLES

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This study produced a highly concentrated gold solution using nanoparticles (NPs) synthesized from sugarcane bagasse as an adsorbent for gold(III) recovery from an aqua regia gold leached lean solution. A 100 kg sample of gold ore collected from a mining site in Iperindo was concentrated. Mineralogical characterization and a fire assay test of the ore were carried out. Atmospheric and agitation leaching were conducted using aqua regia. Sugarcane bagasse NPs were prepared and used to study the gold(III) adsorption kinetics. Gold-loaded sugarcane bagasse NPs were desorbed in a 4 M aqua regia solution. The results revealed that the ore has a low water retention capacity which renders it friable. A microscopic examination established the presence of gold particles and pyrite in the ore. Ore leaching was eminently favoured by agitation yielding a weight loss of 6.3 % when an acid concentration of 4 M was used, the temperature was 90 °C and the leaching time was 30 minutes. Synthesized nanocellulose was able to adsorb gold(III) from the leachate optimally with the following process parameters: 14 mL/g of adsorbent, 106.2 minute-long contact time, a temperature of 56 °C and an agitation rate of 45 rpm when the concentration of leachate was 84 mg/L. The adsorption efficiency of the sugarcane bagasse NPs is comparable with that of commercial activated carbon, namely 92 and 95 %, respectively. The study concluded that NPs synthesized from sugarcane bagasse could be effectively used when upgrading gold and in purification processes.

**Keywords:** adsorbent, aqua regia, leaching, agitation, nanocellulose, bagasse, gold, mineralogical

### 1. Introduction

Gold is a precious metal barely constituting about 0.005 parts per million (ppm) in the earth's crust and must be upgraded by a value of about 3500 to obtain commercial concentrates [1]. Gold ore deposits that are refractory contain pyrrhotite and pyrite, impeding their cyanidation treatment if a roasting pretreatment is not carried out beforehand [2]. Cyanidation presents two challenges related to toxicity and its ability to dissolve gold occluded in carbonaceous materials is limited [3].

When refractory gold ore is processed by a cyanidation treatment, both the concentration and quantity of cyanide required are high, resulting in effluents with cyanide concentrations greater than the permissible level of less than 0.1 ppm [4]. Aqua regia is capable of creating a powerful oxidizing environment that can vigorously dissolve both free gold and gold pre-robbed by sulfides and carbonaceous materials [5].

The isolation and concentration of precious metals involve established methods, namely precipitation, ion exchange, solvent extraction and adsorption. Solutions with high concentrations are preferably recovered using precipitation and solvent extraction but adsorption and ion exchange are preferred for solutions containing trace concentrations.

Activated carbon has been extensively applied for gold recovery by adsorption from leached solutions. Gold recovery using activated carbon is usually not quantitative and significant concentrations of gold are usually left in the leachate [6]. Bioadsorbents have been prepared from materials sourced from plants, e.g. polysaccharides, cotton, paper and biomass wastes. Adsorbents generated from these sources have been utilized in gold(III) recovery and are more highly selective when recovering gold compared to other metals [7]. It is highly likely that a nanoparticle (NP) generated from sugarcane (*Saccharum Officinarum*) bagasse will selectively adsorb gold(III) ions more efficiently than the typically used adsorption gels or activated carbon,

probably because the total surface area to volume ratio of the NPs is very high [8].

## 2. Materials and methods

### 2.1. Sample collection

About 100 kg of gold ore was collected from an active mine in the village of Imogbara located in the town of Iperindo, within the local government area of Atakumosa in the vicinity of Ilesha in Osun State, Nigeria. The sample was collected from 2 different pits that were dug over a surface area of 3 x 3 m to a depth of 4 m below the surface. The ore sample collected was pre-concentrated by panning in a flowing stream close to the mining site. The panned ore was mixed to ensure an even distribution following the method carried out by Mallo [9]. Sugarcane bagasse was collected from a local juice shop in the vicinity of Papalanto in Ogun State. The bagasse was washed using distilled water to remove dirt and remnants of sugar.

### 2.2. Sample preparation

The dried sample was crushed using a Pascal Engineering crushing mill with machine number 18862 operated at 415 V, 2200 W, 4.9 A and 50 Hz [10]. Consequently, a working sample of 1 kg was selected using the coning and quartering method once the bulk sample had been adequately mixed before being pulverized using a pulverizer and sieved through a 75  $\mu\text{m}$  mesh sieve. The oversized particles were pulverized again and sieved until 80 % of them passed through. Representative samples of the ore were carefully taken within the size range of 300  $\mu\text{m}$  > X > 75  $\mu\text{m}$  and those X < 75  $\mu\text{m}$  were concentrated using a shaking table with model number 148566Q while adopting previously applied methods [11].

The obtained bagasse was sun-dried to constant weight before being dried in an oven at 105 °C. The dried bagasse was pulverized and sieved through a 125  $\mu\text{m}$  mesh sieve.

### 2.3. Functional group determination

Infrared spectra of the gold ore samples, sugarcane bagasse and sugarcane bagasse NPs were recorded on a PerkinElmer LS-55 Luminescence spectrometer equipped with a standard attenuated total reflectance (ATR) crystal detector. Spectra of each sample were scanned within the wavelength range of 500-4000  $\text{cm}^{-1}$ . About 10 mg of each pulverized sample was used for each test following a previously applied procedure [12].

### 2.4. Agitation leaching of the ore with aqua regia

Agitation leaching of the ore sample was carried out in a conical flask at a temperature of 90 °C using an aqua regia concentration of 4 M on a magnetic stirrer operated at

300 rpm. Leachings over a period of 30 minutes were replicated using 5 g of gold ore sample in a conical flask to which 25 mL of freshly prepared 4 M aqua regia solution was added. The prepared sample was covered with aluminium foil and placed on a magnetic stirrer preset at 90 °C and 300 rpm before the residual solid was separated, dried and reweighed to determine how much mass was lost. The leachate was collected and stored to be used for the adsorption test, while the concentration of gold in it determined using atomic absorption spectrometry (AAS).

### 2.5. Preparation of the sugarcane bagasse NP adsorbent

The sugarcane bagasse NP adsorbent was prepared following a previously applied methodology [13]. The bagasse was washed using distilled water to remove dirt and remnants of sugar before being sun-dried to constant weight and dried in an oven at 105 °C. The dried bagasse was pulverized and sieved through a 125  $\mu\text{m}$  mesh sieve. The dried bagasse was dewaxed in a Soxhlet extractor using n-Hexane over five runs. The dewaxed bagasse was delignified four times using acidified sodium chlorite at 75 °C for 1 hr each to achieve a white product. The product was treated with 2 wt.% KOH at 90 °C for 2 hrs and subsequently treated with 5 wt.% KOH at 90 °C for 2 hrs before being filtered and rinsed until the pH of the residue was neutral. The product obtained was chemically purified cellulose (CPC). About 10 g of CPC was stirred in 50 mL of boiling concentrated sulphuric acid in a beaker at 45 °C for 45 minutes on a magnetic stirrer. The hydrolysed pulp was centrifuged at 10,000 rpm for 15 minutes to remove excess acid and dialyzed against water until a pH similar to that of distilled water was attained before centrifugation was repeated at 10,000 rpm for 15 minutes. The product was then sonicated for 10 minutes in an ice bath to obtain a concentrated aqueous suspension of cellulose nanocrystals (CNCs) which were stored in a refrigerator at 4 °C.

### 2.6. Adsorption of gold cations

Gold cations were adsorbed according to a previously applied methodology [14] using the prepared sugarcane bagasse NPs in an Erlenmeyer flask. About 0.5 g of the adsorbent was mixed into a 20 mL sample of gold leachate containing  $\text{Au}^{3+}$ . The flask was placed on a magnetic hotplate stirrer for various time periods and temperatures, namely 30, 60, 90 and 120 minutes as well as 30, 40, 50 and 60 °C. This procedure was repeated using 0.75, 1.00 and 1.50 g of the adsorbent. Next the phases were separated by filtration and the cation concentration remaining in the leachate measured using AAS. The percentage of cation uptake was calculated using the following equation:

$$\text{uptake \%} = \frac{(C_0 - C_e)}{C_0} \times 10 \quad (1)$$

where  $C_o$  and  $C_e$  denote the initial and equilibrium concentrations of the cations (mg/L), respectively.

## 2.7. Determination of gold content using AAS

The analysis was carried out in accordance with a previously applied method [15] using an AAS model AA320N. The filtrate obtained by using aqua regia to leach a known mass of the ore was transferred to a 250 mL separatory funnel. The beaker was rinsed with 3 M HCl to ensure the leachate was completely transferred into the funnel. About 10 mL of methyl isobutyl ketone (MIBK) was added and the mixture shaken for 5 minutes before the phases were allowed to separate and the aqueous phase was drained off. Another 10 mL of washing solution was measured into the separatory funnel and the mixture shaken for 2 minutes. Once the phases had completely separated, the aqueous phase was drained off. This washing was done repeatedly to ensure the content was completely removed. The organic phase was stored in a glass tube sealed tightly with a lid. The results obtained were used to construct a calibration curve.

## 2.8. Adsorption kinetics

The adsorption kinetics were determined according to a previously applied methodology [16]. Approximately 200 mg of sugarcane bagasse NPs were thoroughly mixed with 200 mL of gold leachate and stirred on a magnetic hotplate stirrer at 30 °C. Samples of approximately 3 mL were taken at regular intervals of 30, 60, 90, 120 and 150 minutes from the start of the experiment which was repeated at 40 and 50 °C. The amount of Au(III) adsorbed on the adsorbent was plotted against the shaking time at different temperatures on graphs.

## 2.9. Surface morphology of the adsorbent

Scanning electron microscopy (SEM) analyses were conducted using a JEOL JSM-7600F model SEM with an acceleration voltage of 15 kV. The prepared sugarcane bagasse NPs were coated to a depth of only a few nanometres using a sputtering coater to avoid the coating interfering with the morphology of the sample before the sample was mounted with a conductive bridge carbon tape, which connected it to the sample holder. Images showing the surface morphology of the nanocellulose were taken at a magnification of 40,000x.

## 2.10. Preparation of a highly concentrated gold solution

A sample of Au-loaded adsorbent was prepared by stirring 5 g of the prepared nanocellulose with 200 mL of the leachate solution containing a gold concentration of 84 mg/L. After becoming saturated with gold, the adsorbent was separated from the remaining liquid by vacuum filtration, washed and dried at 50 °C. Desorption of the gold-loaded nanocellulose sugarcane bagasse was carried out using a 4 M aqua regia solution as the eluent. The mixture was stirred for 3 hrs after which the solid phase was separated from the liquid one. The concentration of gold in the liquid was measured and the desorbed percentage calculated.

## 3. Results and discussion

### 3.1. Ore upgrading

Energy dispersive X-ray fluorescence (EDXRF) analysis of the ore fed through a shaking table yielded a gold concentration of 2.4 g/ton. However, the gold



Figure 1: FTIR spectra of the gold ore samples

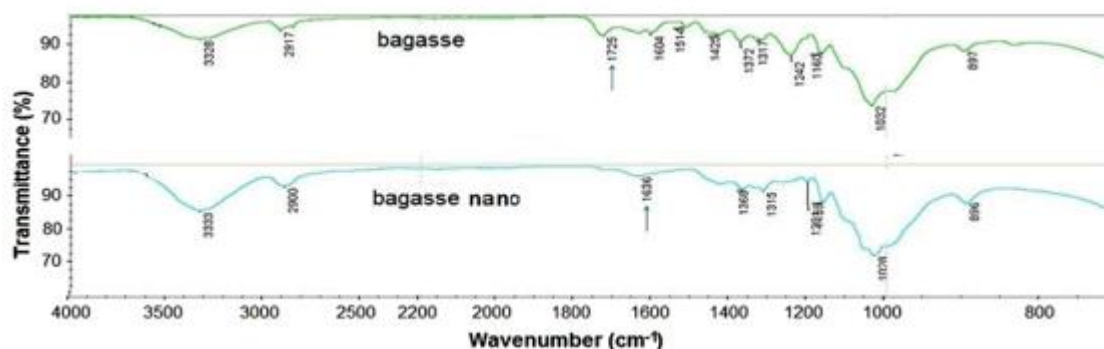


Figure 2: FTIR spectra of sugarcane bagasse and sugarcane bagasse nanocellulose

concentration in the concentrate obtained from the gravity separation was estimated to be 13.6 g/ton. The results obtained showed that the gold ore from Iperindo was significantly upgraded to produce a gold ore concentrate with a high gold concentration.

### 3.2. Functional groups present in the gold ore and sugarcane bagasse

From the Fourier-transform infrared spectroscopy (FTIR) analysis, the results of the gold ore are presented in Figure 1. Various functional groups can be identified in the gold ore. FeS, quartz and SiC are represented by peaks 650.03, 1082 and 777.34  $\text{cm}^{-1}$ , respectively. The presence of these functional groups in the as-received ore suggests the presence of quartz, pyrite, arsenopyrite and silica.

The FTIR spectra of sugarcane bagasse and sugarcane bagasse NPs are shown in Figure 2. Two absorbance bands could be observed ranging from 750-1750 and 2700-3600  $\text{cm}^{-1}$ . The FTIR spectra of the two samples displayed a wide band from 3200-3500  $\text{cm}^{-1}$  indicative of the free O-H stretching vibration of the OH groups in cellulose molecules as suggested by Kumar et al. [13]. The spectra of the two samples reveal the characteristic C-H stretching vibration at approximately 2890  $\text{cm}^{-1}$ .

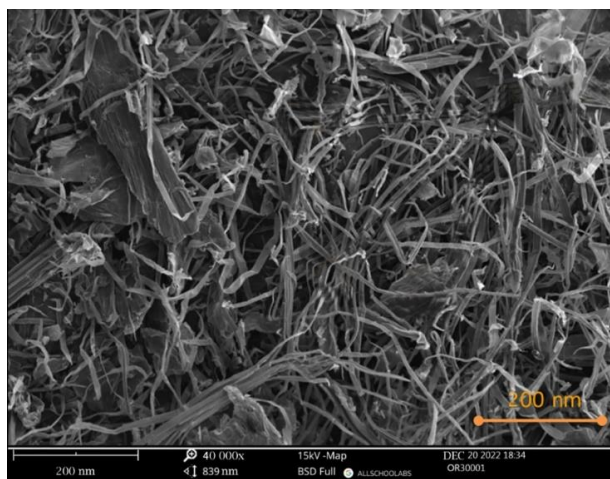


Figure 3: SEM of sugarcane bagasse NPs

### 3.3. Determination of the size of the synthesized nanoadsorbent

Figure 3 shows the SEM micrograph of the synthesized sugarcane bagasse NPs which shows the fibrils of purified cellulose whose diameter and size were significantly reduced due to the entire removal of the amorphous region of semi-crystalline cellulose leaving nanoscale rod-like crystals.

### 3.4. Adsorption kinetics of Au(III)

The data regarding the adsorption kinetics of Au(III) is presented in Tables 1 and 2 as well as illustrated in Figure 4, which shows the variation in the quantity of Au(III) taken up by the nanocellulose synthesized from sugarcane bagasse relative to the shaking time at the

Table 1: Concentration of the leachate after various time intervals (mg/L)

Time (min)	Temperature ( $^{\circ}\text{C}$ )			
	30	40	50	60
0	84	84	84	84
30	53	44	36	59
60	45	34	29	35
90	38	27	21	26
120	32	18	15	19
150	31	18	15	19

Table 2: Percentage of Au(III) uptake after various time intervals

Time (min)	Temperature ( $^{\circ}\text{C}$ )			
	30	40	50	60
0	0	0	0	0
30	37	48	57	50
60	46	60	65	58
90	55	68	75	69
120	62	78	82	77
150	63	79	82	77

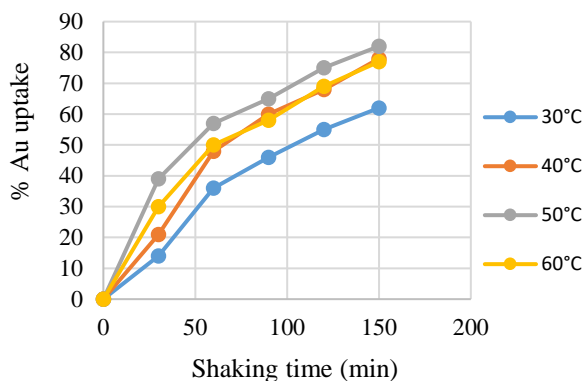


Figure 4: Adsorption rate of Au(III) by nanocellulose synthesized from sugarcane bagasse at different temperatures

different temperatures studied. The initial gold(III) concentration ( $C_o$ ) was 84 mg/L. From Figure 5, it was observed that the temperature influences the adsorption rate of Au(III). The time required to reach the adsorption equilibrium was reduced by increasing the temperature up to 50 °C. A sharp increase in the adsorption rate was noted during the earlier stages of adsorption before decreasing to virtually zero during the final stages. This was observed at all the temperatures examined as a result of the abundant surface area of the nanocellulose available for adsorption during the initial stages which diminished as the period of adsorption advanced.

### 3.5. Highly concentrated gold solution

The obtained gold solution in a 4 M aqua-regia solution was analysed to measure the gold concentration using AAS which was found to be 2200 mg/L. The obtained solution can be effectively processed using electrowinning to obtain metallic gold.

### 3.6. Adsorption isotherms

Data concerning the adsorption equilibrium of Au(III) ions obtained by stirring 10 mg of nanocellulose from sugarcane bagasse with 10 mL of gold leachate of various Au(III) concentrations, namely 10, 20, 30, 40 and 50 mg/L, in an Erlenmeyer flask at a temperature of 40 °C are presented in Table 3, which were fitted to the Langmuir, Freundlich and Temkin adsorption isotherms.

#### Langmuir adsorption isotherm

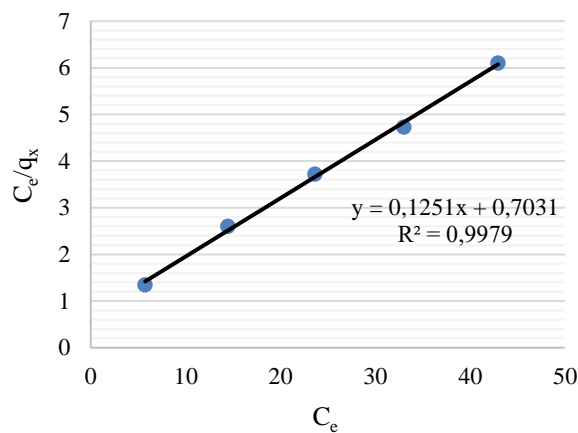
A quantitative description of the monolayer formation of the adsorbate on the outer surface of the adsorbent is given by this isotherm after which further adsorption did not occur, representing the equilibrium spread of the gold ion between the liquid and solid particles.

Equation can be transformed into a linear form to obtain Langmuir adsorption parameters:

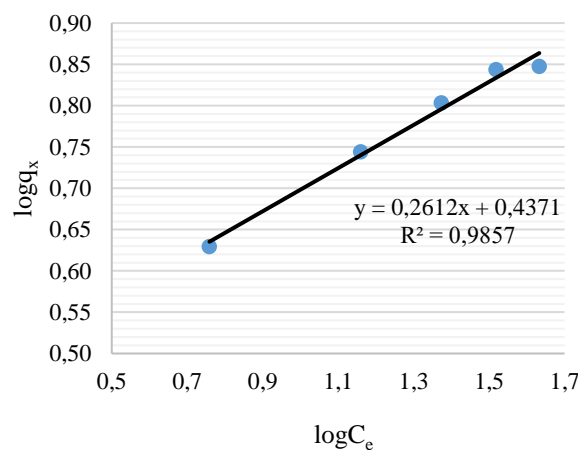
$$\frac{C_e}{q_x} = \frac{C_e}{q_m} + \frac{1}{q_m K_L} \quad (2)$$

where  $C_e$ ,  $q_x$ ,  $q_m$ ,  $K_L$  represent equilibrium concentration, equilibrium adsorption capacity, maximum theoretical

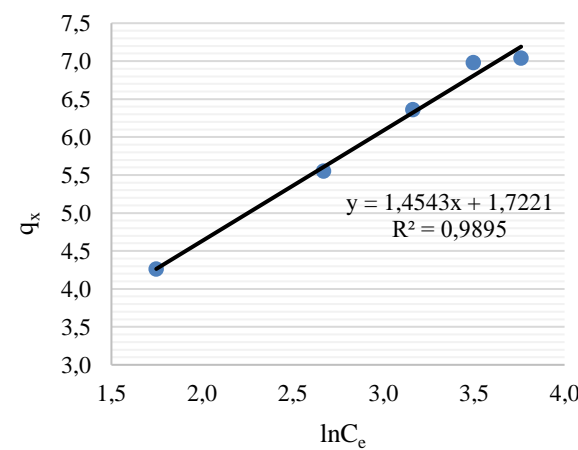
adsorption capacity and Langmuir model constant, respectively.



(a)



(b)



(c)

Figure 5: (a) Langmuir, (b) Freundlich and (c) Temkin isotherms of gold adsorption

Table 3: Parameters for plotting Langmuir, Freundlich and Temkin adsorption isotherms obtained at 40 °C

$C_o$ (mg/L)	$C_e$ (mg/L)	$C_o - C_e$	$q_x = (C_o - C_e) * V/m$	$\log C_e$	$\ln C_e$	$\log q_x$	$C_e/q_x$
10	5.74	4.26	4.26	0.7589	1.7475	0.6294	1.3474
20	14.45	5.55	5.55	1.1599	2.6707	0.7443	2.6036
30	23.64	6.36	6.36	1.3736	3.1629	0.8035	3.7170
40	33.02	6.98	6.98	1.5188	3.4971	0.8439	4.7306
50	42.96	7.04	7.04	1.6331	3.7603	0.8476	6.1023

$q_m$  and  $K_L$  at the respective temperatures were computed from the gradient and intercept of the Langmuir isotherm plot of  $C_e/q_x$  against  $C_e$  (Figure 5a).

The essential characteristic of the Langmuir adsorption isotherm is expressed by a dimensionless separation constant  $R_L$  defined by:

$$R_L = \frac{1}{(1 + K_L C_o)} \quad (3)$$

where  $C_o$  (mg/L) denotes the lowest initial concentration of the gold solution. The  $R_L$  values indicate that adsorption is irreversible ( $R_L = 0$ ), favourable ( $0 < R_L < 1$ ), linear ( $R_L = 1$ ) or unfavourable ( $R_L > 1$  or  $R_L < 0$ ) [17].

#### Freundlich adsorption isotherm

Adsorption onto a heterogeneous surface through a multilayer adsorption mechanism is described by the Freundlich adsorption isotherm. The adsorption is heterogeneous because of the availability of a number of functional groups and numerous adsorbate-adsorbent interactions [18]. The linear form of the Freundlich adsorption isotherm model used for the adsorption is presented in the following equation:

$$\log q_x = \log k_f + 1/C_i \log C_e \quad (4)$$

$k_f$  and  $C_i$  at the selected temperature were determined using the values of intercept and gradient of the linear plot between  $\log q_x$  and  $\log C_e$ , respectively (Figure 5b). Based on standards, when  $1 < C_i < 10$ , adsorption is favourable and the higher  $C_i$  is, the stronger the adsorption intensity [19].

#### Temkin adsorption isotherm

Here the heat of adsorption is assumed to vary linearly as a function of the coverage as opposed to against the logarithmic variation in the case of the Freundlich equation [20]. The equation of the Temkin adsorption isotherm is expressed linearly as follows:

$$q_x = \frac{RT}{\beta} \ln k_t + \frac{RT}{\beta} \ln C_e \quad (5)$$

where  $T$  denotes the absolute temperature (K),  $R$  is taken as the gas constant (0.0083 kJ/mol·K),  $\beta$  represents the Temkin constant which is related to the heat of adsorption (kJ/mol) and  $k_t$  stands for the Temkin isotherm constant (L/g). The parameters  $k_t$  and  $\beta$  at the

temperature considered were estimated by plotting the variation in  $q_x$  against  $\ln C_e$  (Figure 5c).

The parameters derived from the various graphs plotted for the Langmuir, Freundlich and Temkin adsorption isotherms are shown in Table 4. Comparing the  $R^2$  values that correspond to the isotherms evaluated revealed that the adsorption of gold onto sugarcane bagasse nanocellulose obeys the Langmuir adsorption isotherm model preferentially at 40 °C. Furthermore,  $R_L$  calculated for the Langmuir adsorption isotherm model at the three different temperatures considered fell between 0 and 1, strongly indicating that the adsorption of gold was favourable. For the Freundlich adsorption isotherm model,  $C_i$  obtained for the adsorption of gold at the three different temperatures considered fell predominantly between 1 and 10, indicating that adsorption is favourable. However, the values measured were below 4, suggesting a moderate adsorption intensity.

Table 4: Parameters evaluated by using various isotherms for gold ion adsorption onto sugarcane bagasse nanocellulose

Isotherm type	Parameters	40 °C
Langmuir	$q_m$	7.9936
	$K_L$	0.1779
	$R^2$	0.9979
	$R_L$	0.1578
Freundlich	$k_f$	2.736
	$C_i$	3.828
	$R^2$	0.985
Temkin	$\beta$ (kJ/mol)	1.786
	$k_t$	3.268
	$R^2$	0.989

#### 4. Conclusions

Synthesized nanocellulose from sugarcane bagasse using acid hydrolysis was used for the adsorption of gold(III) from the leachate. The effect of the temperature, agitation speed, adsorbent dosage and adsorption time on the rate as well as efficiency of adsorption was studied. At first, the gold uptake increased as a function of the temperature and peaked at 50 °C. However, at 60 °C, the gold uptake capacity of the adsorbent began to drop. The various composite graphs showed that the uptake efficiency increased as a function of the contact time, typically peaking after a contact time of approximately 90 minutes beyond which the adsorption efficiency began to reduce. The gold recovery rate varied as a function of the adsorbent dosage. The adsorption rate as well as percentage recovery initially increased rapidly and gradually reached an equilibrium at dosages of approximately 1.5 g. The gold-loaded adsorbent was desorbed using a 4 M aqua regia solution to yield an Au solution with a concentration of 2200 mg/L, which can be further processed by electrowinning or the precipitation of zinc to obtain metallic gold.

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