OriginalResearchArticle

APPLICATION OF ACTIVATED RICE HUSK FOR ADSORPTIVEBLEACHINGOFGROUNDNUTOIL:KINETIC,EQUILIBRIUMANDTHERMODYNAMIC STUDY

ABSTRACT

Kinetic, equilibrium and, thermodynamic studies of the bleaching of crude groundnut oil at optimizedconditionswerecarriedoutusingactivatedricehuskasanadsorbentforthebleaching process. The efficiency of bleaching wasestimated by measuring the absorbance using a double-beam spectrophotometer at a wavelength of 450nm. The effects of adsorbent dosage, bleaching temperature and, contact time on the bleaching efficiency were studied. A directly proportional relationship wasfoundbetweendosageandcontacttimeandthebleachingefficiencywhileabell curvewas discoveredfortemperatureincrease.Thesurfaceareaofthericehuskincreasedfrom 150.32 to 1450.32 m^2/g while the pore volume decreased from 0.15524 to 0.12844 cm^3/g after activation which was determined via a Brunauer-Emmet-Teller (BET) analysis and the results further validated by the Scanning Electron Microscopy (SEM) images obtained. The kinetic data of the bleaching process were best described by the pseudo-second orderkinetic model while the equilibrium adsorption isotherm analysis showed that the results from the Temkin isotherm were the most significant. The thermodynamic study revealed that the adsorptive bleaching process is feasible,spontaneousand,exothermicwithadecreaseinentropy.Theenthalpyvaluealsoshowed that the adsorption process is predominantly physisorption. This study has revealed that an effective adsorbent can be produced from rice husks under optimized process conditions.

Keywords: KineticModels,TemkinIsotherm,ThermodynamicParameters, Adsorption.

1. INTRODUCTION

A vegetable oil made frompeanut seed that is taxonomicallycategorized as *Arachis hypogaea* is known as groundnut oil, also known as Peanut oil or Arachis oil. This legume crop is mostly farmed foritspalatableseeds.Commercially,therearethreewaystoextract peanutoil: hydraulic pressing, expeller, and solvent extraction. For almost complete oil recovery when hydraulic pressing is utilized, hot solvent extraction is then performed. Oil extraction with an expeller dependsoninternalpressureand frictionthat heatsthemealand makes it easierto extracttheoil. Theamountofpeanutoilremovedwiththisprocedureisabout50%.Hexaneisusedtoextractthe leftover oil, and it is then eliminated via an evaporation-condensation process. Utilization of petroleum hydrocarbons or other solvents is a need for solvent extraction. With hexane, 95% ethanol, or 100% ethanol, this procedure performs more well [1] [2].

Oneofthebyproductsofriceprocessing intherice mills isricehusk. Whenpaddyrice is husked inthefirststepofmilling,thehuskiscreated,anditislaterstrippedfromothercomponentsofthe ricegrain.Asawaste-utilizationresourcethataddsvalueandlowersprocessingcostsinbothhome and commercialsettings, rice huskshave gained popularityasa resource.Asa by-productofrice milling, rice husk is commonly accessible in rice-producing nations like China and India, which produce 33% and 22% of the world's rice, respectively. Between 16 and 25 percent of paddy is madeupofricehusks[3][4][5].Around500 milliontonsofpaddyareproducedworldwideeach

year, yielding 120 milliontons ofrice husk annually [6]. Whenrice husk is burned inanambient environment, a byproduct is produced called rice husk ash. The globe produces 20 million tons annually[7] [5].Ricehusksarenot beingusedproperlysincepeopleareunawareoftheirqualities anduses.Asaresult,theuseofricehusksandricehuskashinhomeandindustrialprocessingnot onlyhelpsto,directlyandindirectly,increasefarmrevenue,but italsooffersanalternatesolution to the problem of how to dispose of rice husks.

Adsorptionisothermsmakepredictionsaboutthequantityofanadsorbatethatwouldbeabsorbed bythesurfaceoftheadsorbent under typicaltime, temperature, andadsorbent usecircumstances. The equilibriumbetweenthe refined oiladsorbatemolecules and the surface ofthe rice husk will bedemonstratedinthisstudy.Amongotherequations,theFreundlichandLangmuirisothermsare employed to characterize the adsorption isotherms. These adsorption isotherms distinguish the adsorptionprocessaccordingtothekindofmolecularinteractionanddescribethesorptionactivity ofthe majority ofadsorbents. The adsorption rate may be calculated by fitting experimentaldata into severalkinetic models, predicting how adsorbent and adsorbate will interact, and simulating theprocess.Pseudo-firstorderorpseudo-secondorderkinetics,amongothers,aregoodpredictors of the rate kinetics for the adsorption process of bleaching groundnut oil.

Thisstudyaimsto clarifythemodeandextentofadsorptiondatausingtheLangmuir,Freundlich, Temkin,andDubinin-Radushlkevichisotherms,aswellasthermodynamicparameters(changein energy, enthalpy, and entropy), to better understand the bleaching mechanism of groundnut oil with a low-cost adsorbent (acid-activated rice husk).

2. MATERIALSANDMETHODS

MATERIALS

Rice husk obtained from Gidan Kwanu village, Bida LGA Niger state was used to prepare the adsorbent. The groundnut oil was purchased from Jemaa LGA, Kaduna state. All the chemical used during the experiments were of analytical grade. The samples (adsorbent and oil) were prepared.Thisincludedtheacidactivationofthericehuskandthedegummingandneutralization of the groundnut oil. The oil was bleached with varying conditions of temperature, adsorbentdosage,andbleachingcontacttime.The best performanceofbleachingefficiencywasnoted,andthe associated parameter chosen as optimum and used to conduct the kinetic, equilibrium andthermodynamic studies. The best performing values from the adsorption characteristics studywere:

Dosage– 8.67g,Temperature–74℃,Contact time – 52 min.

ThepreparedadsorbentwascharacterizedviaBET andSEManalysisaswell.

METHODS

BRUNAUER-EMMET-TELLER(BET)ANALYSIS

The equipment used for the analysis was the Nitrogen BET surface area and pore size analyzer (JW-DA). To determine the surface areaofthe rice husk sample, the BETequationshownbelow was used:

$$\frac{1}{W[(\frac{P_0}{P})-1]} = \frac{1}{W_M C} + \frac{C-1}{W_M C P_O}[\frac{P}{}] \quad (1)$$

Where W is the weight ofnitrogen gas adsorbed at arelative pressure, P/P_0, $W_M$is the weight of adsorbate making up a monolayer ofsurface coverage, and C is the BET C constant. C is related to the energy of adsorption in the first adsorbed layer, so its value shows how strong the interactionsbetweentheadsorbent andtheadsorbateare.TheBETequationneedsalinearplotof $1/[W(P_0/P)-1]$vs.P/P_0.which,whennitrogenisusedastheadsorbate,islimitedtoalowerlimited area of the adsorption isotherm, usually in the P/P_0range of 0.05 to 0.35, because the rice husk sample is microporous.

PREPARATIONSFORSAMPLEANALYSIS

The cold trap Dewar is filled with liquid Nitrogento ¾ of its volume and is then mounted onthe Autosorb-1. The He and N_2cylinder valves as wellas the ball valves which are located after the pressure regulators are opened and the cylinder pressure regulators are set to 10 psi.

DEGASSINGSAMPLESPRIORTOANALYSIS

The right size cell with a fill glass rod (9 mm) was picked for the rice husk sample. The empty sample cell with the full glass rod was weighed and written down on a 5-place analytical scale. The fillglass rodwastakenoutofthe cell, and the rice husk sample was put inusing a tube until it filled up half of the cell. The fill glass rod sample was then put back into the cell. The sample cell,glassstick,andsamplewereallputonascale.Theweightofthesamplewasfoundbytaking theoriginalweight awayfromtheweightofthesamplebefore it wasdegassed. Thecellwasthen putintoaheatingmantleandclampedintoplace.Thecellandheatingmantlewerethenconnected tothesamplepreparationmachine.Theknurledretainerring,plungerwithO-ring,andcelladapter werealltakenoffthesampleprocessing stationand put onthesamplecell. Then, thecellwasput into the hole inthe station, and the nut was lightlytightened. The temperature was set byhand to 200 °C, and the heat was turned on by moving the toggling switch to the "up" position. When degassingstarted,thegreenlight startedblinking. Becauseofhowthesamplewasset up,degassing took 2 hours.

Figure1:BETSurfaceAreaAnalyzer ModelNoJW-DA:76502057en

SCANNINGELECTRONMICROSCOPY(SEM) ANALYSIS

Scanning Electron Microscope Model JOEL-JSM 76000F was the tool used to do the research. The rice husk samples were made by cutting them downto the right size so theywould fit in the specimenchamber.Theywerethenfirmlyattachedtothespecimenholder(alsocalledaspecimen stub).Thesampleswerethencoveredwithalayer ofelectricallyconductiveplatinum, whichwas putonthericehuskbyevaporatingtheplatinuminahighpressure.TheSEMdeviceputthesample in a relatively high-pressure room with a short working distance and a differentially pumped electron optical column to keep the vacuum at the electron gun at a good level. In the ESEM, a high-pressure area around the sample cancels out the charge and boosts the secondary electron signal. This makes the picture of the rice husk sample better at higher magnifications. The low- voltage SEM studywas done ina FEG-SEM because the field emissionguns (FEG) canproduce high primary electron brightness and a small spot size on the rice husk even at low acceleration potentials.

Figure2: Scanning ElectronMicroscopeModelJOEL-JSM76000F

KINETIC,EQUILIBRIUMANDTHERMODYNAMICEXPERIMENTS

To study the adsorption kinetics of activated carbon from rice husk, 30 ml of unbleached Groundnut oil was discharged into the bleaching vessel and bleached at the optimized variables obtainedfromtheRSM.Sampleswerewithdrawnandfilteredforstorageinsamplecontainersfor absorbance analysis.

The procedure was repeated for samples to be withdrawn after subsequent time intervals of 10 minsforthekineticstudy,1.5gfor theequilibriumstudies atfourdifferenttemperaturesand,20 °Cforthethermodynamicstudy.

ADSORPTIONKINETICS

PSEUDO-FIRST-ORDERRATEEQUATION

Thepseudo-first-order equationcanbeexpressedinanon-linear equationasshownbelow

$$q_t = q_e[1 - exp(-k_1 t)] \quad (2)$$

Where,$q_t$and$q_e$areadsorptioncapacityattimetandatequilibriume,respectively,i.e.,theamount of adsorbate per unit of adsorbent and $k_1$is the pseudo-first-order constant t^{-1}[8].

PSEUDO-SECOND-ORDERRATEEQUATION

Thepseudo-second-orderequationcanbeexpressed inanon-linear equationasshownbelow

$$q_t = \frac{q_{e2}^2 k t}{1 + q_e^2 k_2 t} \quad (3)$$

Where$k_2$isthepseudo-second-orderconstantt^{-1}[8].

INTRA-PARTICLEDIFFUSION

TheWeber-Morrisintra-particlediffusionmodelisexpressedas

$$q_t = k_{id} t^{0.5} + C_i \quad (4)$$

Where, k_{id}(mg/kg $min^{0.5}$) istheintra-particlediffusionrateconstant and C_i(mg/kg) isassociated to boundary layer thickness [9].

If intra-particle diffusion is the limiting step of the adsorption process, the plot $q_t$against $t^{0.5}$ is a straightline.Moreover,ifthisplotgoesthroughtheorigin,intra-particlediffusionistheonlyrate-limiting step; if the plot presents two or more intercepting straight lines, adsorption involves independent steps.

ELOVICHMODEL

TheElovich modelcan beexpressed asshown below

$$q_t = \frac{1}{a} \pm \ln(1 + abt) \quad (5)$$

Where, a istheparameter oftheElovichmodelassociatedwiththe initialvelocity($mgkg^{-1}min^{-1}$) b is the desorption constant ($mgkg^{-1}$).

EQUILIBRIUMSTUDIES

The isotherm parameters correlate the kinetics and thermodynamics of the adsorption process, providingaquantitativeandqualitativeestimationoftheefficiencyoftheadsorbent.Inthiswork, fouradsorptionisothermswereusedtoevaluatetheoptimumbleaching byadsorptionofgroundnut oil using activated rice husk as an adsorbent.

LANGMUIRISOTHERMMODEL

TheLangmuir modelwasthe first modelpresentingacoherenttheoryofadsorption. It assumes a monolayer surface coverage, an independent and homogenous adsorbent surface, mostlyapplied to chemisorption. It is generally expressed in a non-linear model as shown below.

$$q_e = \frac{q_{max} K_S C_e}{1 + K_S C_e} \quad (6)$$

Where, q_e(mg/kg) is the amount of adsorbate per unit mass of adsorbent, C_e(mg/kg) is the equilibrium concentration of adsorbate in solution and q_{max}(mg/kg) and K_S(mg/kg)$^{-1}$ are Langmuir constants related to the adsorption capacity and rate of adsorption for the monolayer, respectively[10].

FREUNDLICHISOTHERM MODEL

TheFreundlichmodelis anempiricalone, assuminga heterogenoussurfaceenergy, i.e., stronger binding sites are occupied first, and the binding strength decreases with an increasing degree of siteoccupation,andit isnotrestrictivetomonolayercoverage.Itisgenerallydescribedinitsnon- linear form as shown below.

$$q_e = K_F C^n{}_e \quad (7)$$

Where,K_F[(mg/kg)(mg/kg)$^{-n}$]isdefined astheadsorptioncapacityoftheadsorbent[10].

TEMKINISOTHERM MODEL

Alinearformofthe TemkinIsothermexpression:

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e \quad (8)$$

Where $A_T$is the Temkin isotherm equilibrium binding constant (L.mol^{-1}), $b_T$is the Temkin isotherm constant (J/mol), R is the Universal Gas Constant (J.mol^{-1}.K^{-1}) and T is the absolute temperature (K).

DUBININ-RADUSHLKEVICHISOTHERMMODEL

AlinearformoftheDubinin-RadushkevichIsothermexpression:

$$\ln q_e = \ln q_D - 2B_D RT \ln(1 + 1/C_e) \quad (9)$$

$$E_D = \sqrt{1/2B_D} \quad (10)$$

Where $B_D$relatesto the free energyofadsorption per mole ofcoloredoilpigment inthe solution asit movestothesurfaceoftheadsorbent fromaninfinitedistance (mol^2.kJ^2),$q_D$istheDubinin–Radushkevich isotherm constant, which relates to the degree of sorbate sorption on the sorbent surface (mg.g^{-1}), $E_D$is the apparent energy of adsorption (kJ.mol^{-2}).

THERMODYNAMICSPARAMETERS

TheStandardGibbsFreeEnergycanprovidethedegreeofexoergicityandthehigher itsabsolute value reflects more energetically favourable adsorption. Whereas enthalpy is important to determine whether the adsorption process in chemicalor physical, as it is shown in Equation 11

$$\Delta G^0_{ads} = -RT\ln(K_0) \quad (11)$$

Where, ΔG^0_{ads}(J/mol) is the standard Gibbs Free Energy, R is the universal gas constant (8.3145 $J \cdot mol^{-1} \cdot K^{-1}$), T (K) isthetemperature and K_0isthe equilibriumconstant (orthesolutecoefficient of distribution between the solid and liquid phases at equilibrium) which changes with temperature.

StandardGibbsFreeEnergymightbeexpressedintermsofstandardenthalpy(ΔH^0)and standardentropy(ΔS^0_{ads})accordingtoVan'tHoffequation(12)

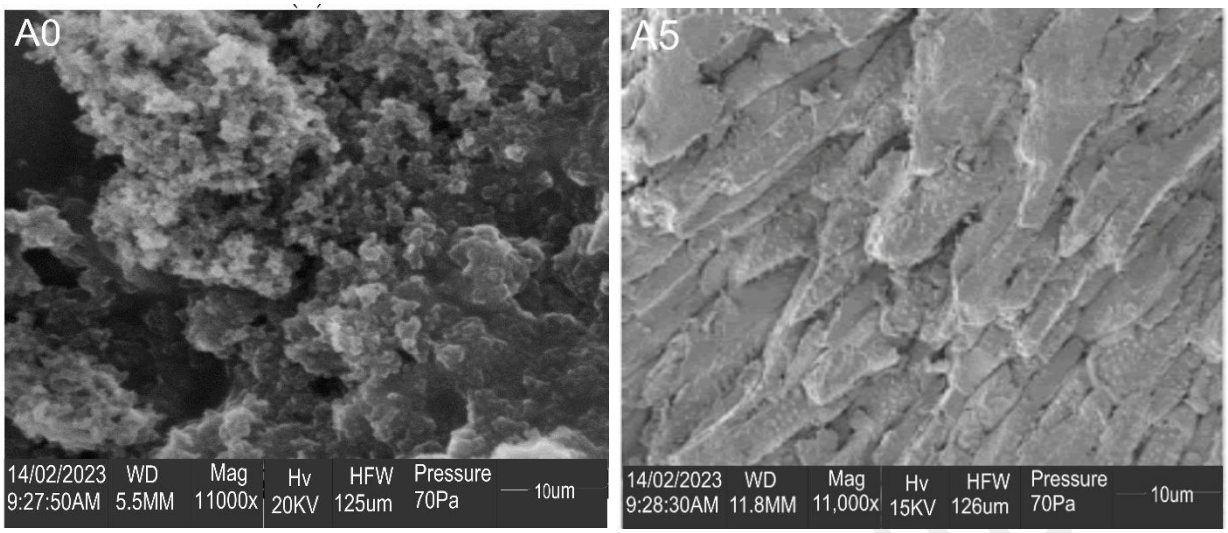
$$\ln(K_0) = -\frac{\Delta G^0_{ads}}{RT} = \frac{\Delta S^0_{ads}}{R} - \frac{\Delta H^0_{ads}}{RT} \quad (12)$$

3. RESULTSANDDISCUSSION

SCANNINGELECTRONMICROSCOPY(SEM)ANALYSISOFTHEADSORBENT

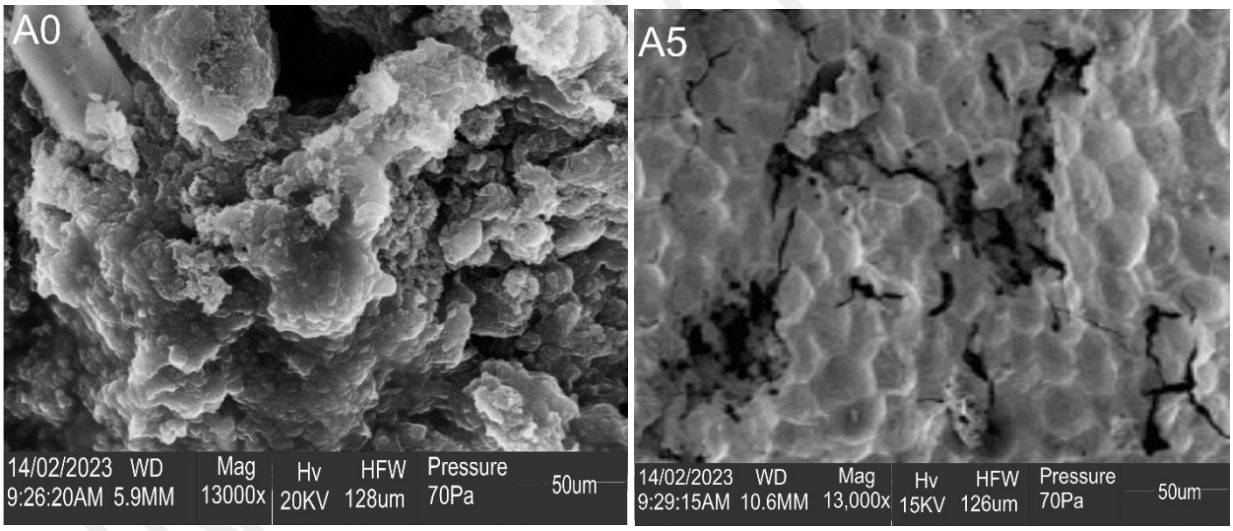
Presented in the figures below are the scanning electron micrographs of raw rice husk and the activated rice husk at various magnifications. The SEM images were studied for the surface and morphologicalcharacteristicsoftheadsorbentmaterials.Incomparisonwiththerawricehusk,the SEMimagesshowedclearchangesinthemicrographsofallthe activatedricehusks.Thischange in the SEM images revealed the structural changes in the modified adsorbent materials.

The images indicated uneven surfaces and non-uniform structures in both samples. The micrographs of the raw rice husk show structures that are few and relatively large with some smalleragglomeratessituatedontheirsurfaceand thestructuressituatedinaporousmatrixspace withlarge interstitialporespaces. This is inagreement withthetexturalcharacterizationobtained via BET analysis that showed the micropore volume of the raw rice husk being greater than the activated rice husk. The micrographs of the activated rice husk show smaller, more numerous structuresthataresmoother,flatterwithouttherandomdispersionofagglomeratesontheirsurface andwelldistributedintheir matrixcreatingamuchlargersurfaceareawhich isfurtherconfirmed bytheBETanalysisthatshowsan864.82%increaseinsurfaceareaoftheactivatedricehusk.The structures are also mildly more uniform tending to "filamentous" and "flake-like" shape that is boththinandelongated,withabranchingorthread-likeappearancethat isalsosomewhatflatand irregular in shape. Changes in adsorption effectiveness between the two materials may be explained by morphological variations between the particles making them up; for instance, the adsorbent that was subjected to acid treatment displays a higher surface area of contact.



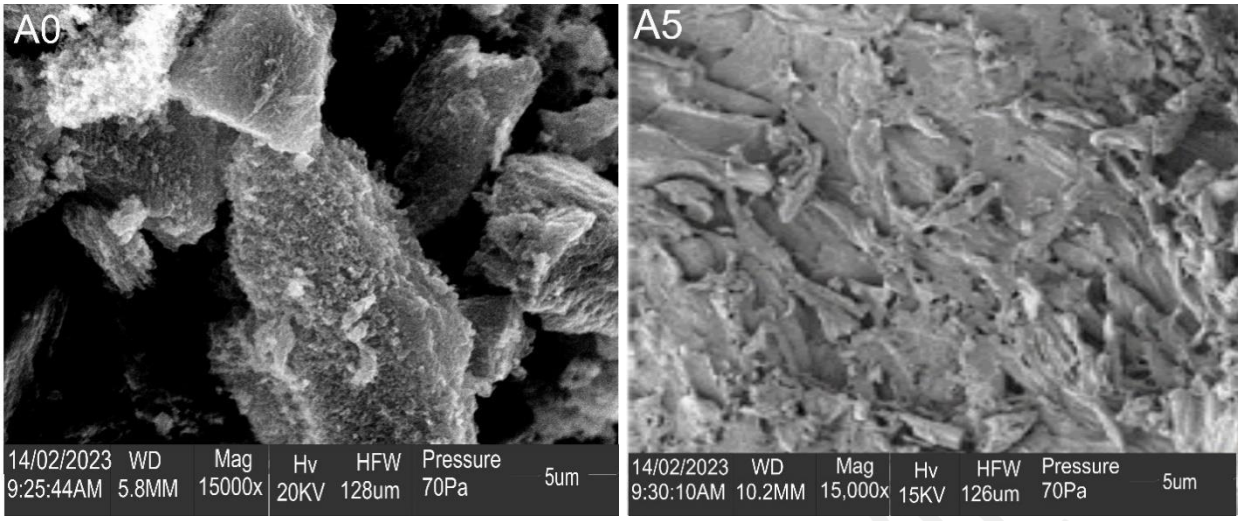
(a) (b)

Figure3:Scanningelectronmicrographsofraw(a)and3MH_2SO_4activated(b)rice husk at 11000x magnification.



(a) (b)

Figure4:Scanningelectronmicrographsofraw(a)and3MH_2SO_4activated(b)rice husk at 13000x magnification.



(a) (b)

Figure5:Scanningelectronmicrographsofraw(a)and3MH_2SO_4activated(b)rice husk at 15000x magnification.

BRUNAUER-EMMETT-TELLERANALYSIS

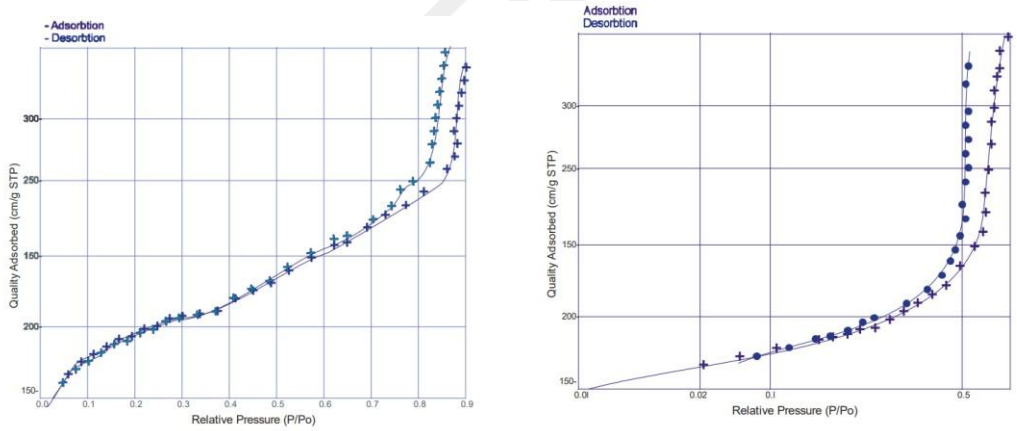
Table 1 summarizes the BET characterization ofthe adsorbents (crude and activated) used in the bleaching procedure. The activated rice husk has a much larger surface area than that of the raw ricehuskwithapercentageincreaseof864.82%.Asreportedby[11],theBETsurfaceareaofraw rice husk was found to be 320 m^2/g and this increase in surface area may be because of acid activation. The BET surface area of1450.32 m^2/g achieved also matches works done by[12] [13] and [14] whom prepared adsorbents from coconut shell, palm kernel shell and rice husk and achieved a BET total surface area of 2,451, 1,135 and 2,696 m^2/g respectively.

Themicroporevolumeandaverageporewidthwerebothgreaterinrawricehuskthaninactivated husk. The scanning electron micrographs (SEM) of these two adsorbents corroborate this. It's possible that a greater quantity of oil will be impregnated at the conclusion of the adsorptive processiftheporespacesandvolumearebigger,sincethisfacilitatesoilentryandincreasesaccess ofthe adsorbateto the adsorption sites inside the pore cavities [15]. The linear and log isotherms ofthe BET analysis for the crude and activated rice husk are presented in the figures below.

Table1: SummaryoftheBETanalysisofthecrudeand activatedricehusk.

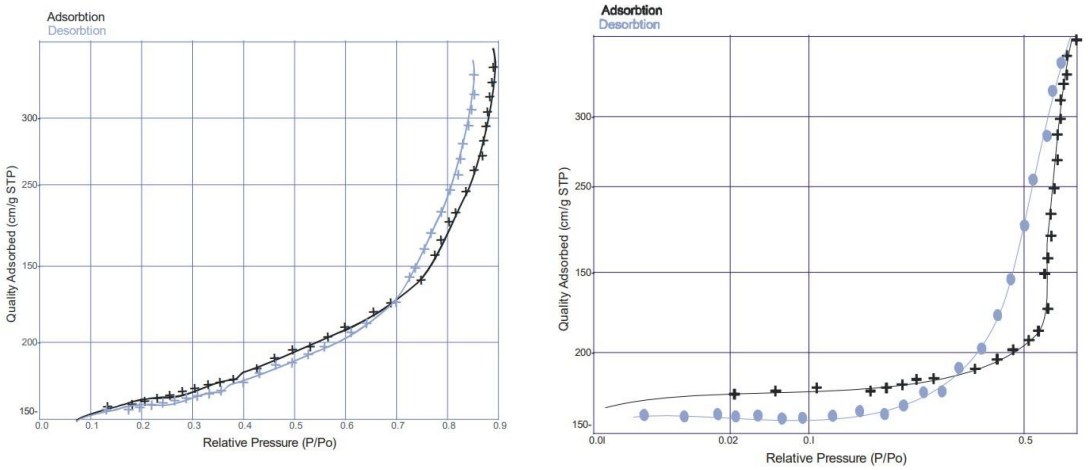
Parameter	**CrudeRice Husk**	**ActivatedRice Husk**
BETTotalSurfaceArea (m^2/g)	150.32	1450.32
t-PlotMicro-poreArea(m^2/g)	50.12	50.23

PoreVolumeofporeslessthan 1550.529Å width (cm^3/g)	0.5545	0.6035
PoreVolumeofporeslessthan 1226.916Å width (cm^3/g)	0.60734	0.60548
t-PlotMicroporevolume(cm^3/g)	0.15524	0.12844
BJHDesorptioncumulativevolumeofpores	0.45222	0.52222
BETAdsorption averageporewidth(4V/A) (Å)	28.25	24.54
BETDesorptionaverageporewidth(4V/A) (Å)	28.22	24.42
BJHAdsorptionaverageporewidth(4V/A) (Å)	28.55	24.26
BJHDesorption averageporewidth(4V/A)(Å)	30.55	24.22



(a) (b)

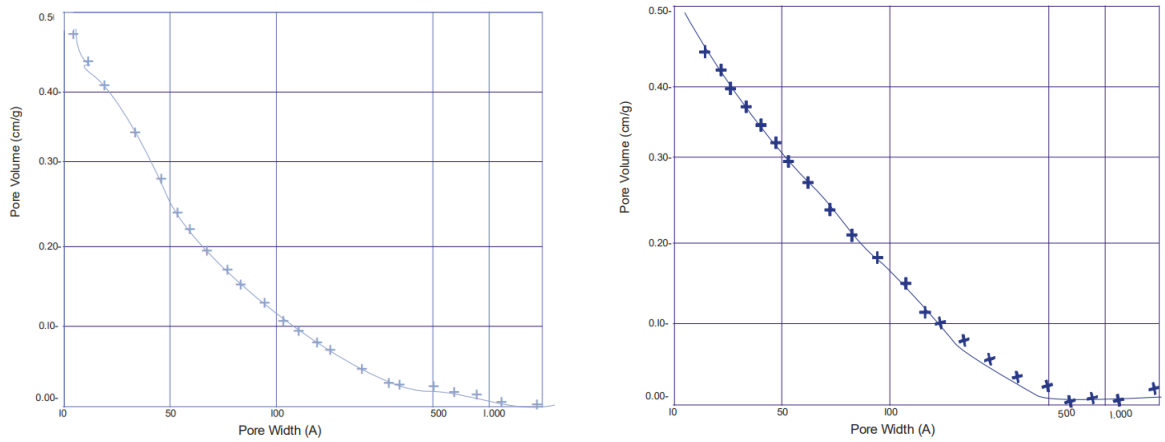
Figure6: NitrogenadsorptionBETlinear (a) andlog(b) isothermplotofactivatedricehusk



(a) (b)

Figure7:NitrogenadsorptionBET linear (a) andlog(b)isothermplotofrawricehusk

Figures8 showtheplotsoftheBJHadsorptioncumulativeporevolume fortherawandactivated rice husk. The aggregate pore volume of a solid substance is shown graphically as a function of pore diameter in a plot known as the BJH (Barrett-Joyner-Halenda) plot. The determination of pore size in adsorbents is an important metric that provides valuable information regarding the adsorption capacity of said adsorbents [16]. Adsorption isotherm data for the material are commonlyacquiredatcryogenictemperaturesbygasadsorptionmethodslikenitrogenadsorption, and the plot is created by fitting the BJH theory to this data. A porous material's pore size distribution may be determined via its adsorption of isotherm values using the BJH theory. It assumesthattheporesinthesubstancesarecylindricalorslit-likeandthattheadsorbedgasatoms create a monolayer on the outermost layer of the substance.



(a) (b)

Figure8:BJHadsorptionporevolumeplotfortheraw(a)andactivatedricehusk(b)

ADSORPTIONCHARACTERISTICSOFTHEBLEACHINGPROCESS

EFFECTOFADSORBENTDOSAGEONTHEBLEACHING PROCESS

Table2:Absorbanceand bleachingefficiencyatvaryingadsorbentdosage

Dosage(g)	Absorbance(A450)	BleachingEfficiency(%)
2.67	0.631	34.41
4.17	0.558	41.20
5.67	0.512	46.78
7.17	0.493	48.75
8.67	0.471	51.04

To studythis effect, adsorption was performed at different adsorbent dosages within the range of 3.67-8.67g at constant optimum temperature, time and, particle size (54 ℃, 22 min and, 0.2 mm respectively).Thecrudegroundnutoilhasanabsorbanceof0.962.AsmaybeseeninFigure9,the bleachingpercentageroseastheadsorbent dose increased. It canbededucedthat theadsorbent dosehasanimmediateimpacton thebleachingprocess,withmoreadsorbentleadingtomore activeadsorptionsitesand hence morebleaching power.It is importantto notethat increasing the doseresultsinarapidimprovementinefficiency,butthattheimprovementinbleachingefficiency tends to levelout at a certain point. Increasing the amount ofadsorbent used in the groundnut oil bleaching process is crucial for optimizing production.

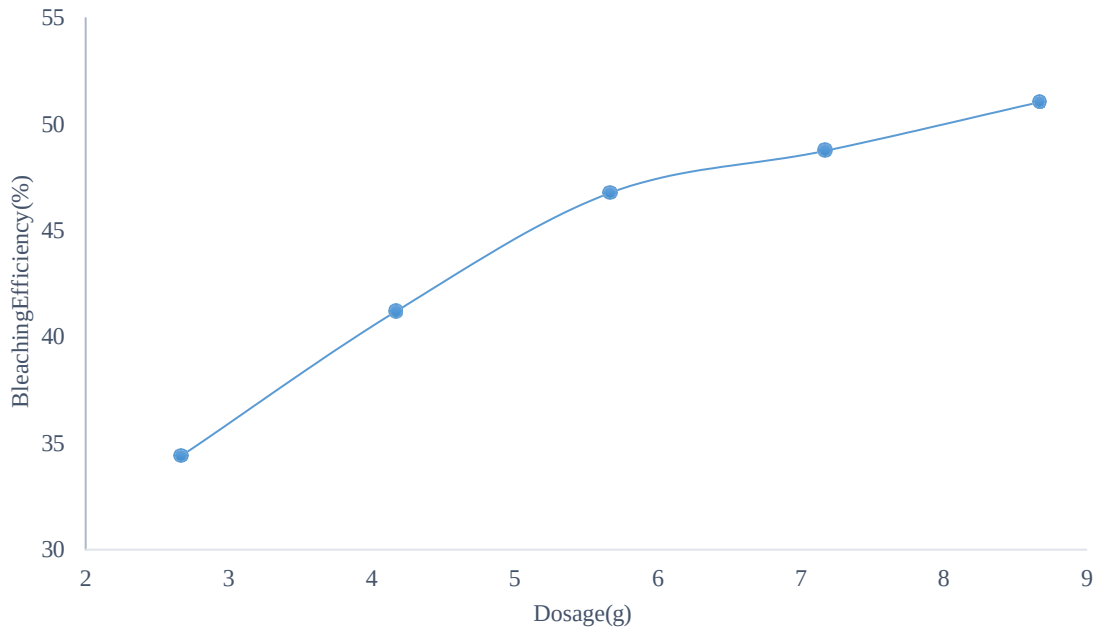


Figure9:Plotofbleachingefficiencyagainstadsorbentdosage

EFFECTOFTEMPERATUREONTHEBLEACHINGPROCESS

Table3:Absorbanceand bleachingefficiencyatvaryingtemperatures

Temperature(℃)	Absorbance(A450)	BleachingEfficiency(%)
34	0.621	35.45
54	0.512	46.78
74	0.502	47.81
94	0.537	44.18

To study this effect, adsorption was performed at different temperature within the range of 34– 94 ℃at constant optimum adsorbent dosage, time and particle size (5.67 g, 22 min and 0.2 mm respectively).Theabsorbanceofthecrudegroundnutoilwasmeasuredas0.962.Figure10 shows that with increased temperature, the bleaching efficiency initially increases until a certain point at which it then begins to decrease. It can be inferred that the bleaching temperature has a directeffecton the bleaching process and theincreasein bleaching power occurs as a resultof higherenergyandadsorptionsitesprovidedbythetemperatureincreaseandthedecreasein bleachingpowermay beasaresultof adestruction of theseadsorption sitesdue tothehigher temperatures. Bleachingtemperatureisanessentialaspect ofthebleachingofgroundnutoilas its rise enhances the effectiveness of the process till a certain point.

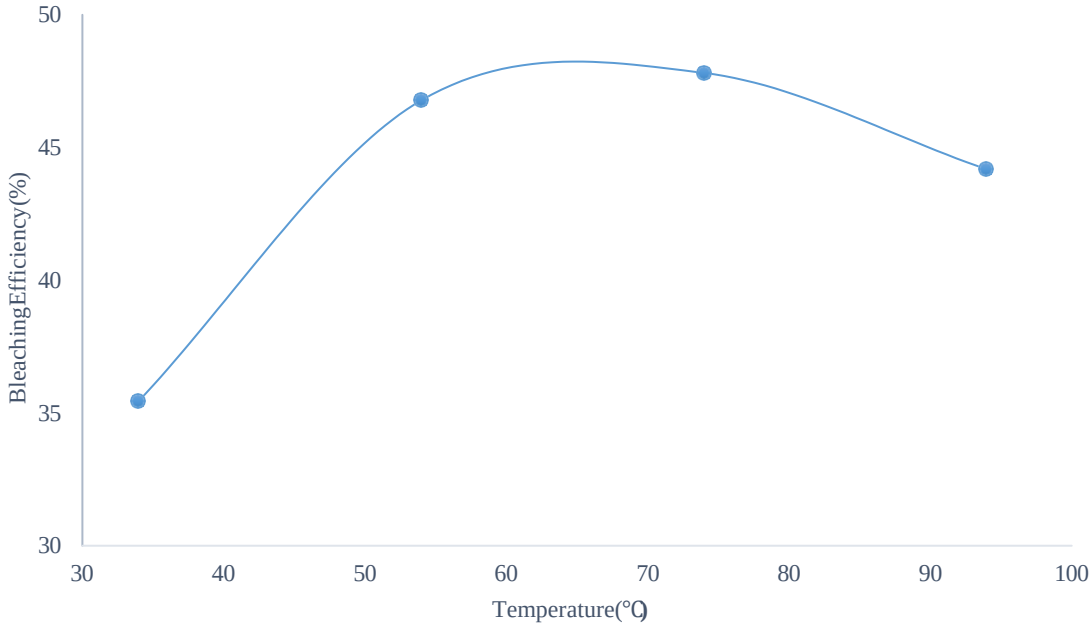


Figure10:Plotofbleachingefficiencyagainsttemperature

EFFECTOFCONTACTTIMEONTHEBLEACHING PROCESS

Table4:Absorbanceandbleachingefficiencyatvaryingcontacttime

Contact Time (min)	Absorbance (A450)	BleachingEfficiency(%)
12	0.667	30.67
22	0.512	46.78

32	0.485	49.58
42	0.472	50.94
52	0.468	51.35

To study this effect, adsorption was performed at different contact time within the range of 12 – 52minatconstantoptimumtemperature,adsorbentdosageandparticlesize(54℃,5.67gand0.2 mm respectively). The absorbance of the crude groundnut oil was measured as 0.962. Figure 11 showsthatthepercentagebleached increasedwithincrease incontacttime.It canbe inferredthat the contact time has a direct effect onthe bleaching process and the increase in bleaching power occurs as a result ofan increase in adsorption ofthe adsorbates to the active adsorption sites due tothelongertimeofcontactbetweenthem.Itshouldbenotedthataninitialincreaseinthecontact time leadstoasteepincrease intheefficiencyandfurtherincrease inthetimetendsto plateauthe bleaching efficiency increase. Contact time is an important factor of the bleaching of groundnut oil as its increase improves the efficiency of the process.

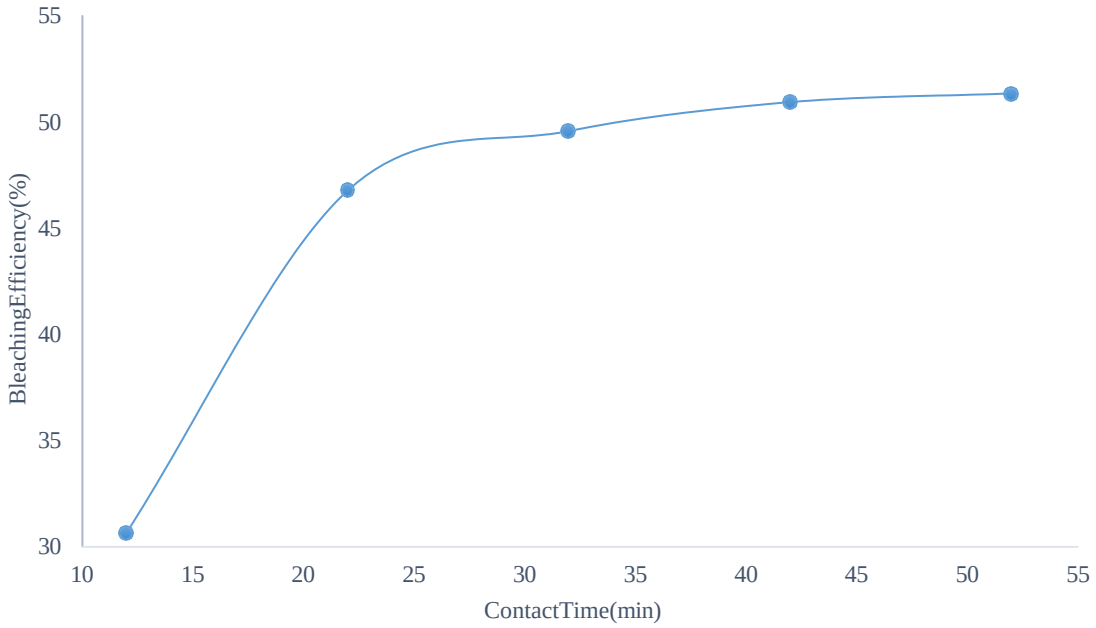


Figure11:Plotofbleachingefficiencyagainstcontacttime

KINETICSTUDIES

Thesoluteabsorptionrateofaprocessmaybedeterminedbystudying itssorptionkinetics. Itisa crucialfactorindeterminingthesuccessofanadsorptionprocess[17].Table5displaysthekinetic parameters calculated from the linear plots of the corresponding kinetic equations. In a head-to-head comparison of the analysed data, the pseudo-second-order model (R^2 = 0.9957) was shown to better characterize the adsorption under the optimal circumstances.

Table5:Kineticparameters fortheadsorptionbleaching process

Kinetic Model	Constants/Parameters		
	$K_1(min^{-1})$	$q_e(mg.g^{-1})$	R^2
Pseudo-FirstOrder	0.0112	0.3034	0.6532
	$K_2(g.mg^{-1}.min^{-1})$	$q_e(mg.g^{-1})$	R^2
Pseudo-Second Order	1.2013	0.4557	0.9957
	$K_d(g.mg^{-1}.min^{-1})$	$\varepsilon(mg.l^{-1})$	R^2
Intra-Particle Diffusion	0.0724	0.9262	0.9357
	$\alpha\times10^{-5}(mg.g^{-1}.min^{-1})$	$\beta(mg.g^{-1})$	R^2
Elovich	9.5008	7.4906	0.8566

PSEUDO-FIRSTORDERKINETICMODEL

ThelinearizedPseudo-firstorderrateexpressionis givenas:

$$\ln(q_e-q_t)=\ln q_e-K_1t \quad (13)$$

Where $q_e$and $q_t$are the amounts of adsorbates at equilibrium time and at and time t respectively (mg/g),K_1isthePseudo-firstorderadsorptionrateconstant(min^{-1}),andtisthecontacttime(min).

Beer lambert'slawgivesadirect relationbetweenconcentrationandabsorbance,sotheequation can be written in terms of absorbance instead of concentration as follows

$$\ln(A_0-A_t)=\ln A_0-K_1t \quad (14)$$

Where: A_t is the absorbance of the oil bleached at a time t and $A_0$is the absorbance of crude or unbleached oil.

The Pseudo-first order at the optimal temperature was expressed by a plot of In (A_0- A_t) vs t, as shown in the figure below; $A_0$and K_1were calculated from the slope and the intercept, respectively.Itwasfoundthatthevalueofthecorrelationcoefficient(R^2)was0.6532.Itwas

discovered that the theoretical $q_e$did not match up well with the experimental q_e. Adsorption is statedtoberegardednottohaveobeyedthekineticmodel,evenifthecorrelationcoefficient$R^2$is rather high, if the estimated values of $q_e$are not verynear to the observed value of q_e[18].

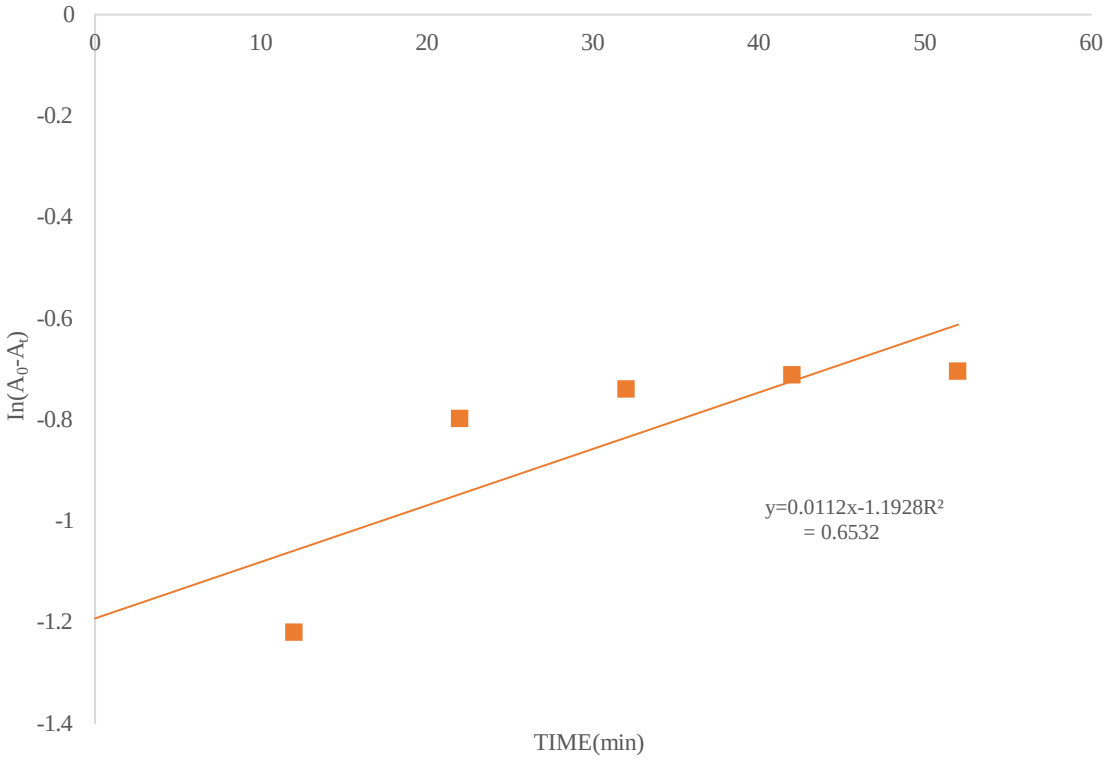


Figure12:Pseudo-firstorderkineticmodelplot

PSEUDO-SECONDORDERKINETIC MODEL

Thelinearformofthepseudo-secondorderexpressionis:

$$\frac{t}{q_t} = \frac{1}{Kq_{2e}^2} + t\left(\frac{1}{q_e}\right) \quad (15)$$

Where$q_e$and$q_t$aretheamountsadsorbedatequilibriumtimeandatanytimetrespectively(mg/g), K_2isthePseudo-secondorderadsorptionrateconstant(g/mgmin),andtisthecontacttime(min).

Beer lambert'slawgivesadirect relationbetweenconcentrationandabsorbance,sotheequation can be written in terms of absorbance instead of concentration as follows

$$\frac{t}{A_t} = \frac{1}{KA_{2e}^2} + t\left(\frac{1}{A_t}\right) \quad (16)$$

Where:A_t istheabsorbanceoftheoilbleached atatimet

Theslopeandinterceptof theplot of t/A_tvstin Figbelowwereusedtocalculate thePseudo- second order rate constant K_2, and A^t for the adsorbent at the optimal temperature.

The adsorbent has an extremely high value of R^2 (0.9957 to be exact). This indicates that the adsorbent is highly correlated with the bleaching process. Equally impressive was the closeness between the estimated and actual values of the equilibrium adsorption capacity (q_e). Since these adsorbents are effective in bleaching palm oil, the Pseudo-second-order model may be reliably applied to this process.

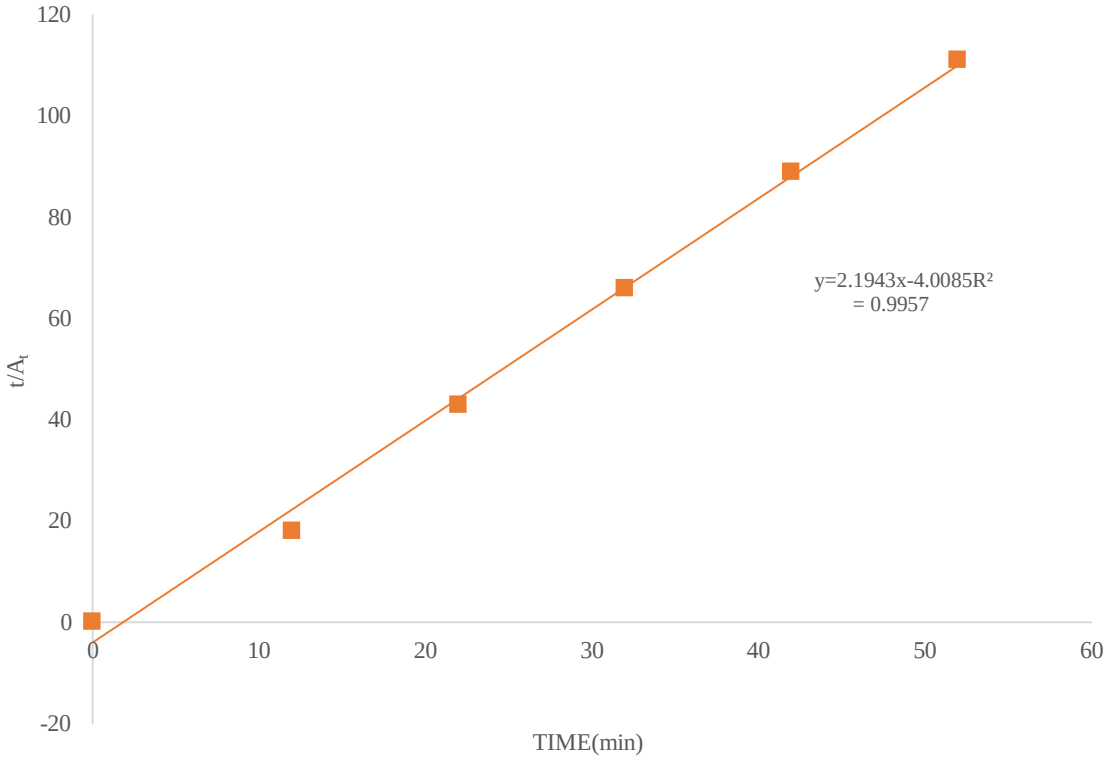


Figure13:Pseudo-secondorderkineticmodelplot

INTRA-PARTICLEDIFFUSIONKINETICMODEL

The most commonly used technique for identifying the mechanism involved in the adsorption processistheintra-particlediffusionplotwhichexpressestherelationshipbetweentheadsorption capacity(q_t) at time $t^{1/2}$. The intra-particle diffusion equation is as expressed below:

$$q_t = K_d t^{0.5} + \varepsilon \quad (17)$$

Where $q_t$is the amount adsorbed at time t (mg/g), $K_d$is the rate constant of the intra particle transport(g/mg/min), ε istheequilibriumconcentrationassociatedwithboundarylayerthickness (mg/l), and t is the contact time (min).

Beer lambert'slawgivesadirect relationbetweenconcentrationandabsorbance,sotheequation can be written in terms of absorbance instead of concentration as follows

$$A_t = K_d t^{0.5} + \varepsilon \quad (18)$$

Where:A_t istheabsorbanceoftheoilbleached atatimet.

Theslopeandintercept of the A_t against $t^{0.5}$ plotwereusedtocalculatetheintra-particlediffusion rateconstants, K_d and ε, fortheadsorbentattheoptimaltemperature.Intra-particlediffusionisthe only rate-limiting mechanism if the linear plot of A_t against $t^{1/2}$ is a straight line from the origin [19]. Inabatchprocess,theadsorptionofathinlayerofsolute ontoasolidsurfacefromtheentire volumeofthesolution istherate limiting step [20]. Dueto thechange in therateofmasstransfer from the first to the last stage of adsorption, the linear lines did not intersect at the origin. This suggeststhatintra-particletransportisnottheonlyrate-limitingprocess,asseenbythelinearlines' departure fromthe origin. Although, the calculated correlation coefficient R^2 was relatively high at a value of 0.9357.

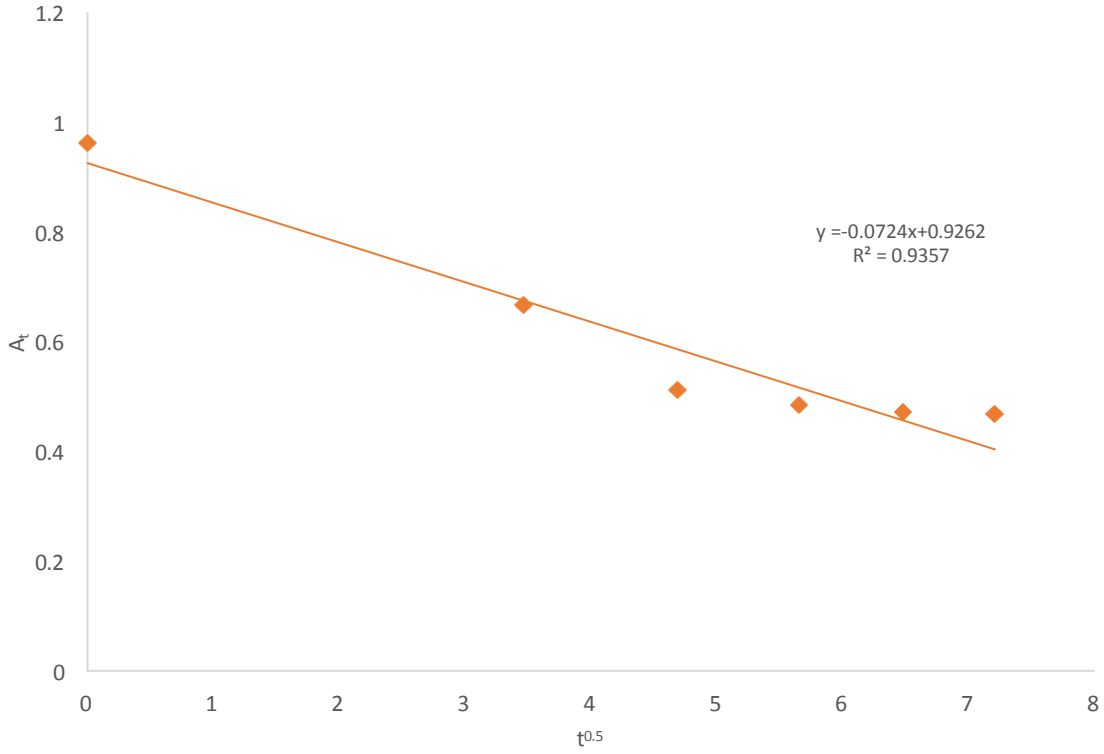


Figure14:Intra-particlediffusionkineticmodelplot

ELOVICHKINETICMODEL

The linearformoftheElovichkineticmodelisexpressedbelow:

$$q_t=(1/\beta)\ln(\alpha\beta)+(1/\beta)\ln(t) \quad (19)$$

Where, α is the parameter of the Elovich modelassociated with the initial velocity($mg.g^{-1}min^{-1}$) and β is the desorption constant ($mg.g^{-1}$).

Beer lambert'slawgivesadirect relationbetweenconcentrationandabsorbance,sotheequation can be written in terms of absorbance instead of concentration as follows

$$A_t = (1/\beta)\ln(\alpha\beta) + (1/\beta)\ln(t) \quad (20)$$

Where: A_t is the absorbance of the oil bleached at a time t.

The Elovich model rate constants, α and β for the adsorbent at optimum temperature were determined fromtheslope and intercept oftheplot of $A_t$against In(t).Thecalculated correlation coefficient of R^2 was found to be 0.8566.

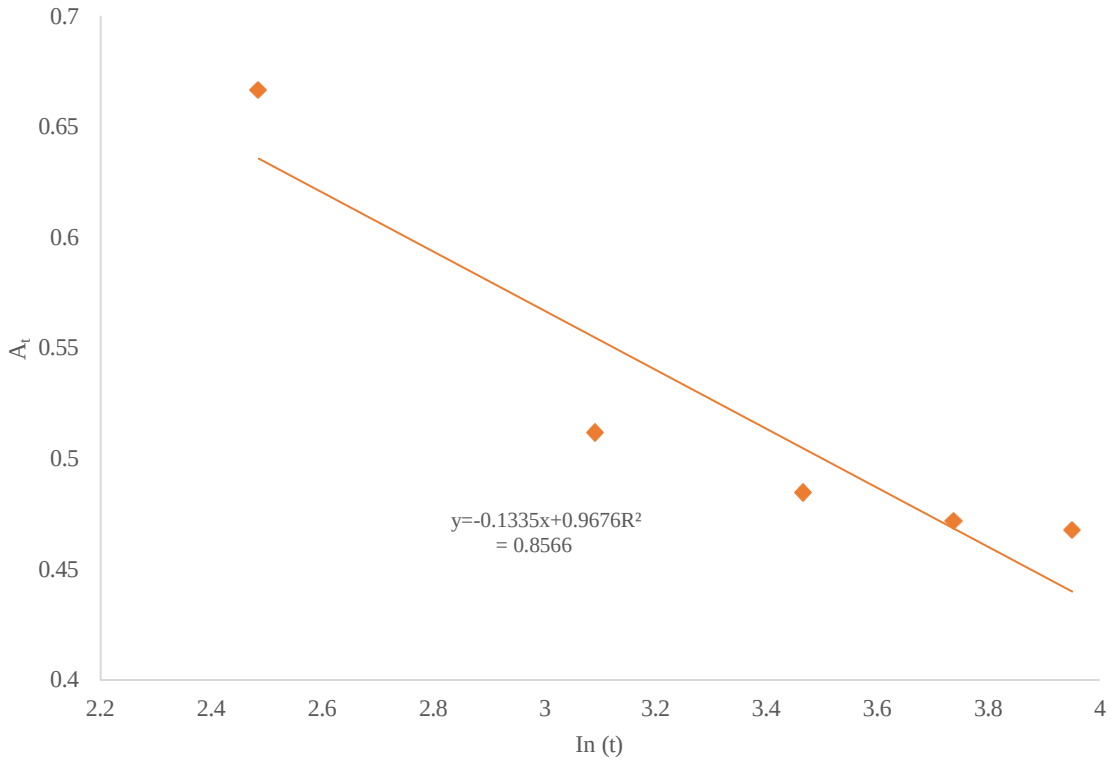


Figure15:Elovichkineticmodelplot

EQUILIBRIUMANDISOTHERMSTUDIES

The adsorption isothermdescribes the connection between the concentration ofthe solute in the liquid phase and the quantityof adsorbent mass needed to remove the solute, held constant throughouttimeandtemperature.To constructaneffectiveadsorptionsystem,one must first get the adsorption isotherm [21]. The adsorption capacityofactivated rice husk for bleaching crude groundnut oilwas calculated using manyadsorption isothermmodels for optimalunderstanding of the adsorption process. The Temkin isotherm was shown to be more favorable to the adsorption process than the other isotherms by comparing their correlation coefficient (R^2) valuesafterthe linearizationandchartingofthe linearstraight-lineconnections.Thisshownthat temperature is very influential in the energy required for sorption processes. Certain energy parameters were revealed via the Temkin and Dubinin-Radushkevich constants. Because the Dubinin-Radushlkevich Isotherm's deviations are not predicated on ideal assumptions like eqi-potentialofsorption sites, lack ofsteric hindrances between sorbed and incoming particles, and surface homogeneityonthe microscopic level, it is the most universalofthe isotherms. Table 6

belowshowsthe isothermparametersfortheconsideredisothermsatthevarioustemperatures they were studied.

Table6:Adsorptionisothermparametersforthebleachingprocess

Isotherm Model	**Parameter/ Constant**	**Temperature(℃)**			
		34	54	74	94
Freundlich	K_F	0.1670	0.4379	0.8638	0.7291
	1/n	2.3298	2.6550	3.5196	3.6108
	R^2	0.9292	0.9721	0.9287	0.9849
Langmuir	K_L	0.8717	1.1397	1.3438	1.3073
	q_m	0.0461	0.0519	0.0359	0.0318
	R^2	0.7607	0.8713	0.7945	0.9770
Temkin	$b_T \times 10^3$	7.6599	5.1024	3.8480	4.2475
	A_T	2.3726	2.7168	2.5418	2.4134
	R^2	0.9532	0.9948	0.9737	0.9508
Dubinin-Radushlkevich	$B_D \times 10^{-4}$	7.5677	7.5691	9.3988	9.1413
	q_D	2.2516	6.4165	29.2820	27.4600
	E_D	25.7041	25.7018	23.0648	23.3874
	R^2	0.9317	0.9762	0.9347	0.9832

FREUNDLICHISOTHERM

A linearformofthe FreundlichIsothermexpression:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (21)$$

Where$q_e$isthenumberofadsorbatesatequilibriumtime(mg/g),$K_f$istheFreundlichequilibrium constant which signifies adsorptive capacity (mg/g), and $C_e$represents the equilibrium concentration of mixture ($mg.L^{-1}$)

The adsorption process ofpigments and oxidation products was tracked using absorbance values accordingtotheLambert-BeerLaw,whichstatesthat absorbance valuesaredirectlyproportional to the concentration of the molecule in the solution.

Thus,

$$C \propto Abs_t = X_e, q \propto (Abs_0 - Abs_t)/m = x/m \quad (22)$$

where Abs_0isthe initialabsorbance ofthe solution (beforeadsorption), Abs_tisthe absorbance of thesolutioninequilibriumafteradsorption,andmistheamountofadsorbentused(g).Therefore, the isotherms q vs C were replaced by the following relationship:

x/mvs X_e

Therefore,theFreundlichisothermbecomes:

$$\text{Log}x/m = \log K_f + \frac{1}{n} \log X_e \quad (23)$$

Where x/m is the amount of adsorbate per unit mass of adsorbent (mg/g), $X_e$is the solute equilibriumconcentrationofadsorbate(mg/l). Therefore,theFreundlichisothermwasstudied by plottinglogx/mversus$\log X_e$,as seeninthefigurebelow.Boththeinterceptandtheslopewere usedtogettheFreundlichconstants$K_f$and$\frac{1}{n}$.Adsorptioncapacityisquantifiedbytheconstant K_f,whereasadsorptionstrengthorfavourabilityisquantifiedbytheconstant$\frac{1}{n}$.Thevalueof$\frac{1}{n}$ willbebetweenzeroandtenforfavourableadsorption[22].In thisstudy,theactivatedricehusk wasshowntohaveapositiveeffectonthecrudegroundnutoil,with$\frac{1}{n}$valuesrangingfrom2.3298 to 3.6108. Adsorption fit the Freundlich isotherm model, as shown by the R^2 values between 0.9287and0.9849.The$K_f$valuerepresentstheabilityoftheadsorbenttoremovecolourfromany givensolute.$K_f$valuesrosewithincreasingtemperature,showinganincreaseintheavailabilityof adsorption sites, but fell marginally at the maximum temperature of 94 °C, suggesting that even this high heat might be damaging to the adsorption sites.

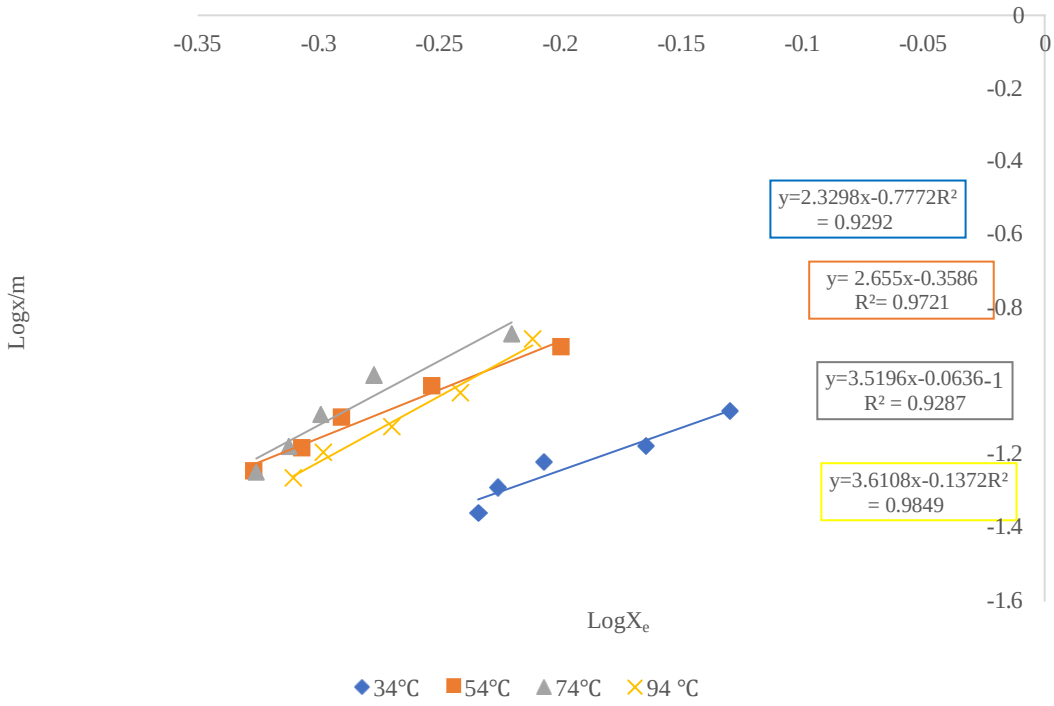


Figure16:Freundlichisothermplotforthebleachingprocessatdifferenttemperatures

LANGMUIRISOTHERM

AlinearformoftheLangmuirIsothermexpression:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L C_e} \qquad (24)$$

Where $K_L$is the Freundlich equilibriumconstant which signifies adsorptive capacity (mg/g) and $q_m$is the theoretical isotherm saturation capacity ($mg.g^{-1}$)

Therefore,theLangmuirisothermbecomes:

$$\frac{X_e}{x/m} = \frac{1}{q_m K_L} + \frac{1}{q_m} X_e \qquad (25)$$

The Langmuir constants $K_L$and $q_m$were calculated bydetermining the intercept andthe slope of the linear plot of $X_e/(x/m)$ vs $X_e$in the figure below. R^2 values for the treated Rice husk varied from 0.7607 to 0.9770. This demonstrates that the adsorption is a close fit to the Langmuir isotherm. It's possible that the uniform arrangement of active sites on the activated rice husk is responsible for the good match between the Langmuir isotherm and the experimental results.

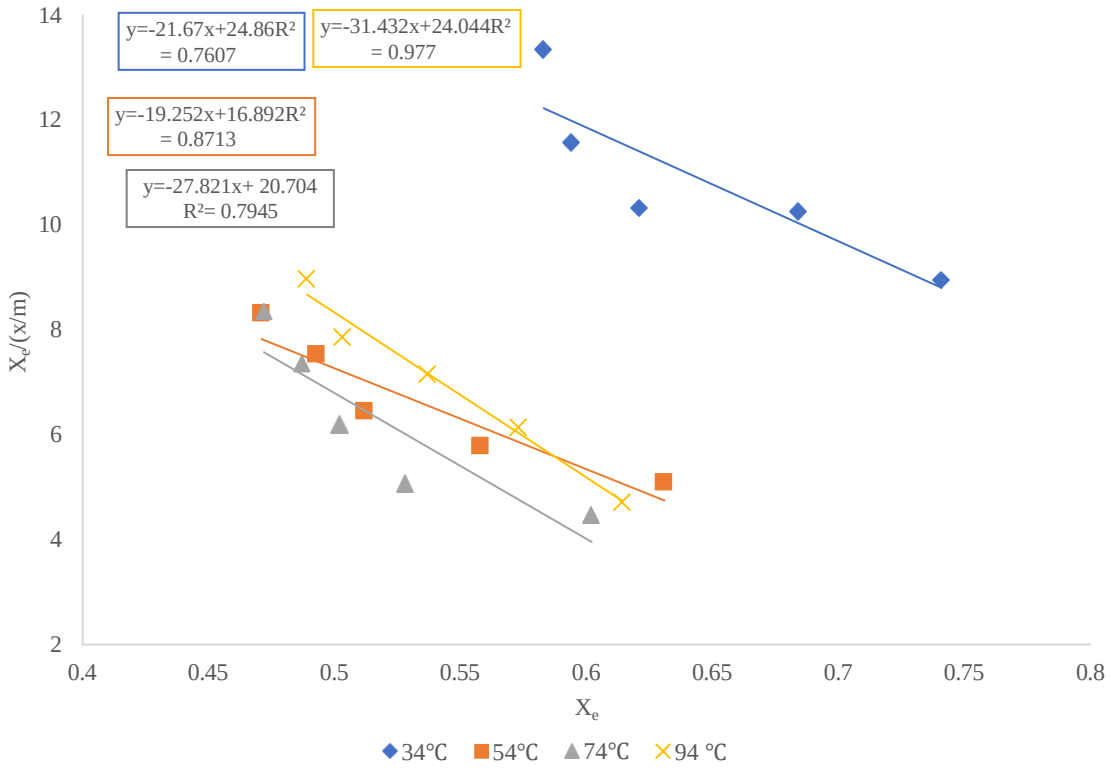


Figure17:Langmuirisothermplotforthebleachingprocessatdifferent temperatures

TEMKIN ISOTHERM

Alinearformofthe TemkinIsothermexpression:

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e \quad (26)$$

Where $A_T$is the Temkin isotherm equilibrium binding constant ($L.mol^{-1}$), $b_T$is the Temkin isotherm constant (J/mol), R is the Universal Gas Constant ($J.mol^{-1}.K^{-1}$) and T is the absolute temperature (K)

Therefore,theTemkinisothermbecomes:

$$\frac{x}{m} = \frac{RT}{b_T} \log A_T + \frac{RT}{b_T} \log X_e \quad (27)$$

For the Temkin isotherm, the values of x/mwere plotted against Log $X_e$in the figure below and the Temkin constants $A_T$and $b_T$were obtained fromthe intercept and slope of the plot.

The Temkin model is often used to illustrate surface energy systems with a heterogeneous composition. Temkin isothermequilibrium binding constant and heat of sorption are denoted by the constants AT and b_T, respectively. Adsorption was shown to be represented by a linear connection in the plot.

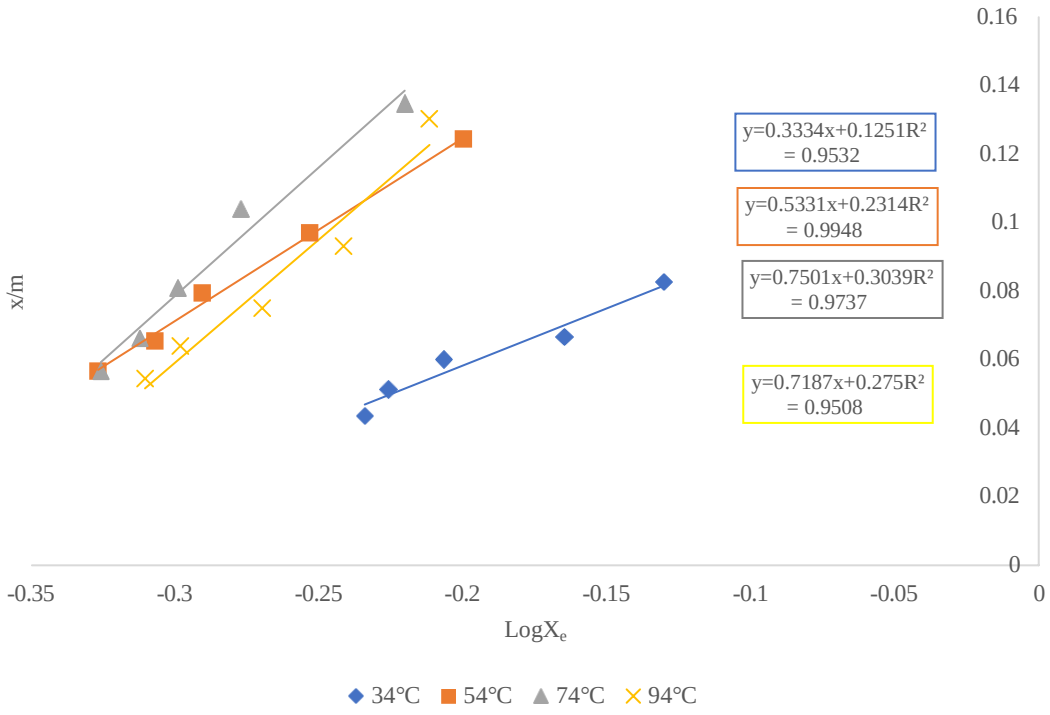


Figure18:Temkinisothermplotforthebleachingprocessatdifferent temperatures

3.5.4DUBININ-RADUSHKEVICHISOTHERM

AlinearformoftheDubinin-RadushkevichIsothermexpression:

$$\ln q_e = \ln q_D - 2B_D RT \ln(1+1/C_e) \quad (28)$$

$$E_D = \sqrt{1/2B_D} \quad (29)$$

WhereB$_D$relatestothefreeenergyofadsorptionpermoleofcolouredoilpigment inthesolution as it movestothesurfaceoftheadsorbent fromaninfinitedistance (mol^2.kJ^2), q$_D$istheDubinin–Radushkevich isotherm constant, which relates to the degree of sorbate sorption on the sorbent surface (mg.g^{-1}), E$_D$is the apparent energy of adsorption (kJ.mol^{-2}). The adsorption process of pigments and oxidation products was tracked using absorbance values according to the Lambert-BeerLaw,whichstatesthatabsorbancevaluesaredirectlyproportionaltotheconcentrationofthe molecule in the solution.

Therefore,theDubinin-Radushkevichisothermbecomes:

$$\ln \frac{x}{m} = \ln q - 2BRT \ln(1+1/X) \quad (30)$$
$$D \qquad D \qquad e$$

FortheDubinin-Radushkevichisotherm,thevaluesofLogx/mwereplottedagainstLog $(1+1/X_e)$in the figure below. Using the graph's intercept and slope, we can get the constants B$_D$and q$_D$. Apparent energy of heterogeneous surface systems is the primary application of the concept. The plot's linearity also revealed the sort of adsorption relationship present on the adsorbents. Chemisorption was detected by an E$_D$value greater than 8 kJ.mol^{-1}.

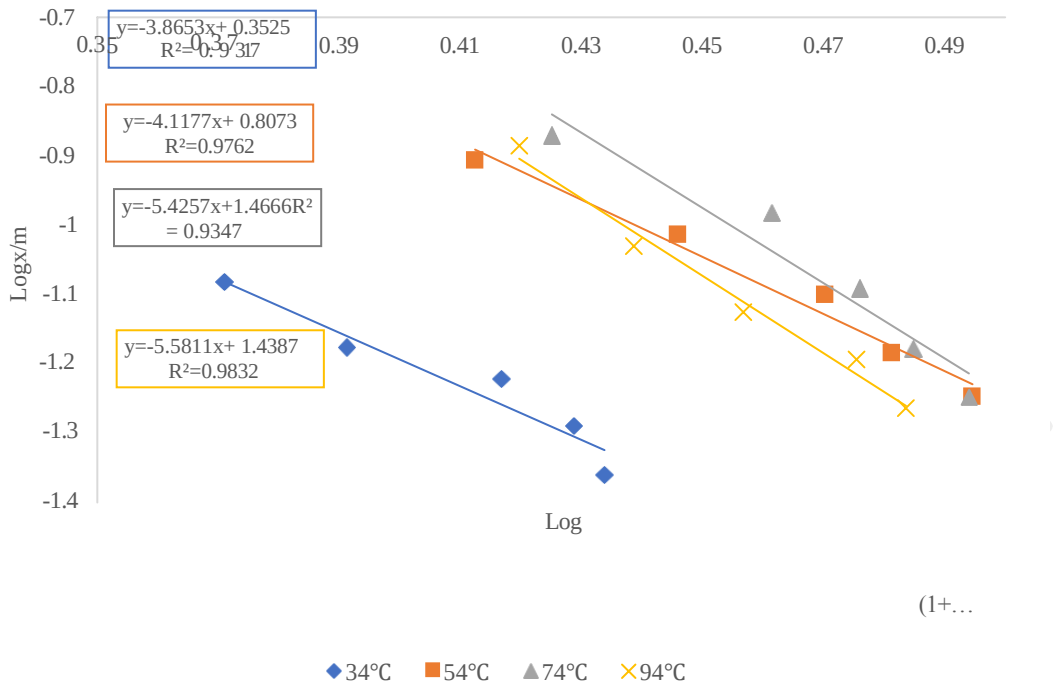


Figure19:Dubinin-Radushkevichisothermplotforthebleachingprocessat different temperatures

THERMODYNAMICSTUDIES

Changesinfreeenergy(ΔG^0),enthalpy(ΔH^0),andentropy(ΔS^0)wereusedtoassess thermodynamicattributes.Anincreaseorreductionintheunpredictabilityoftheprocessatthe solid/solutioninterfaceisdeterminedbyΔS^0,whereasΔG^0 controlswhetherornottheprocess isviableandspontaneous.IfΔG^0isnegative,thenreactionstakeplacespontaneouslyat agiven temperature[23].Sorptiondistributioncoefficientoftheisothermwiththebestmatchmaybeused toquicklyandeasilycalculatethethermodynamicparametersfromdataacquiredfromadsorption isotherm models. This situation calls for the use of the Temkin isotherm. The free energy of the sorption process was calculated by plugging the $A_T$values into the following equation:

$$\Delta G^0_{ads} = -RT\ln A_T \quad (31)$$

Where $\Delta G^0_{ads}$is the free energy of sorption (kJ/mol), T is the temperature in Kelvin and R is the universal gas constant (8.31414 J/mol K). Below is the Van't Hoff equation for the sorption distributioncoefficientasafunctionoftemperatureintermsoftheenthalpychangeΔH^0andthe entropychangeΔS^0:

$$\ln(A_T) = \frac{\Delta S^0_{ads}}{R} - \frac{\Delta H^0_{ads}}{RT} \quad (32)$$

WhereΔH^0 istheheatofadsorption(kJ/mol)andΔS^0 isthestandardchangeinentropy (kJ/mol). A larger negative value for the free energy represents a more energetically favourable sorptionprocess,whichinturnshowsthedegreetowhichthesorptionprocessoccurs

spontaneously. Slopeandinterceptofa1/TvslnA$_T$scatterplotwereusedtocalculateΔH^0_{ads} and ΔS^0_{ads}respectively. (Fig. 20).Table7displaystheresultsofthecalculations. Table 7:

Thermodynamic parameters for the bleaching process

Temperature(K)	ΔG^0_{ads} (KJ/mol)	ΔH^0_{ads} (KJ/mol)	ΔS^0_{ads} (J/mol)
307	-2.2053	-2.9014	-0.5787
327	-2.7174		
347	-2.6914		
367	-2.6882		

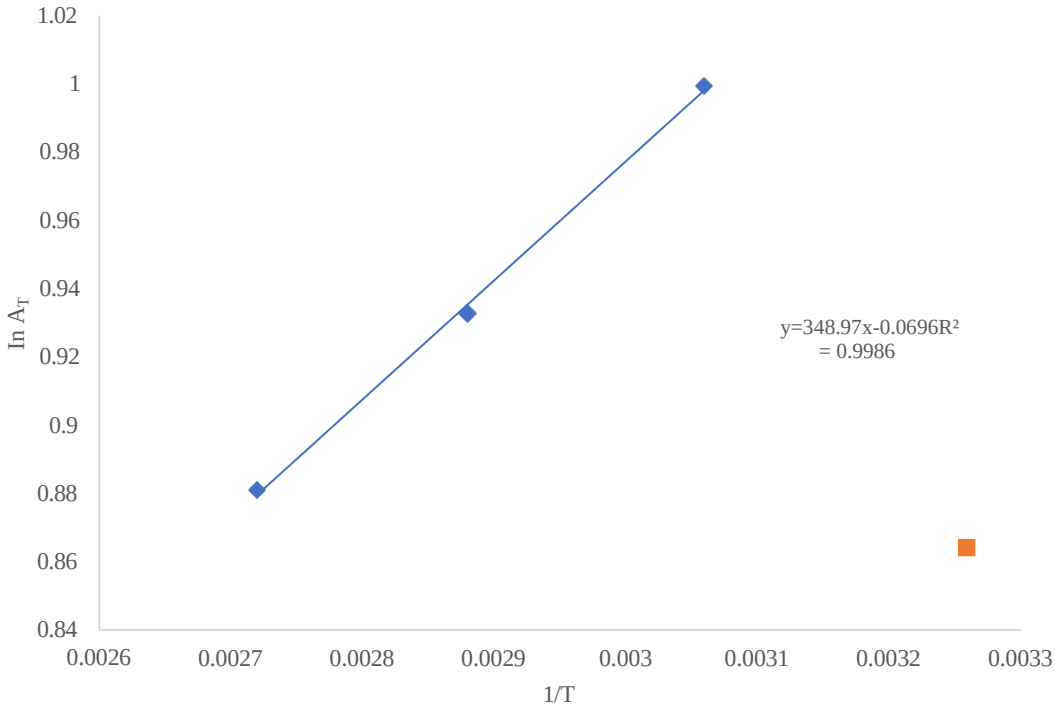


Figure20:Thermodynamicplotforthebleachingprocess

As canbe seen inTable 7, all ΔG^0_{ads}values are negative, andthe degreeofnegativitygrows with rising temperature. Since $\Delta G^0_{ads}$is negative, adsorption bleaching occurs spontaneously and is technicallyfeasible.Thisdemonstrates thatgreatertemperaturesfacilitatethebleachingprocess. Itistruethattheenthalpyofadsorption,ΔH^0_{ads},andnottheentropyofadsorption,ΔG^0_{ads},dictates

thetypeofadsorption,yetvaluesofΔG^0_{ads} intherangeof2.2053-2.7174kJ/molaretypicalof physicaladsorptionsystems.

Since ΔH^0_{ads}isnegative, energyis being givenoffthroughouttheadsorptionprocess;thus, this is anexothermic reaction. Giventhat $\Delta H^0_{ads}$is less than20 KJ/mol, adsorption maybe classified as aphysisorptionprocess.Sincedisorderisrelatedtoentropy,anegativevalueofΔS^0_{ads}impliesless order, i.e., the crude oil has less colorant and fewer contaminants after the bleaching process.

4. CONCLUSION

The kinetic, equilibrium and thermodynamic study of groundnut oil bleaching using adsorbent developedfromactivatedRicehuskbeeninvestigatedinthiswork.ABETanalysiscarriedouton thecrudeandacidactivatedricehuskshowedasurfaceareaincreasefrom150.32m^2/gto1450.32 m^2/g which was further validated by the SEM micrograph images which showed significantmorphologicalchangestotheadsorbent.Thisindicatesthattheactivationprocesshadasignificanteffectonthesurfaceareaavailable foradsorption. Theadsorptioncharacteristicsofthebleaching process showed that adsorbent dosage, bleaching temperature and contact time are all important parameterstobeconsideredduringthebleachingprocess.Thebestconditionsfromthestudywere Dosage – 8.67g, Temperature – 74℃, Contact time – 52 min. The kinetic data of the bleaching process were best described by the pseudo-second order kinetic model while the equilibrium adsorption isotherm analysis showed that the results from the Temkin isotherm were the most significant. The thermodynamic study revealed that the adsorptive bleaching process is feasible, spontaneous, exothermic with a decrease in entropy. The enthalpy value also showed that the adsorption process is predominantly physisorption. This study has revealed that an effective adsorbent can be produced from rice husk under optimized process conditions.

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