Facile Uptake of Cadmium (II) from Aqueous Solution Using Polyamidoamine Functionalized Silica

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Abstract
An amine terminated G 5.0 Polyamidoamine dendrimer was functionalized with Succinic anhydride and immobilized on chromatography grade silica gel to produce a functionalized dendrimer-silica composite (G-5-PAMAM-SGA). The composite was characterized using Fourier Transform-Infra Red spectroscopy, Brunauer Emmett and Teller surface area analysis, Thermogravimetric Analysis, Zeta Potential, and Scanning Electron Microscopy. Adsorption properties of the synthesized composite were studied by batch adsorption method using Cd (II) as adsorbate. The sorption process was analyzed using 6 different isotherm models (Langmuir, Freundlich, Harkins-Jura, Sips, Jossens and Baudu).

The experimental data were best fitted into Jossens isotherm model ($R^2=0.981$) the order of fitting for the models is Jossens>Baudu>Harkins-Jura>Sips>Freundlich>Langmuir. The maximum adsorption capacity of G-5-PAMAM-SGA for Cd (II) was 123.4 mg g$^{-1}$ and the system was influenced by temperature, initial concentration and pH. Analysis of sorption kinetics reveals that the data fitted better to the pseudo-second-order model than any other kinetic model, signifying that the sorption process involved is chemisorption. Sticking probability ($S^*$=0.9999), suggesting a mixture of chemisorption and physisorption (coordination adsorption). Gibbs free energy of adsorption at 293,303 and 313 K were all negative thus sorption was spontaneous and favorable.

Keywords: Polyamidoamine; Succinic anhydride; Isotherm; Chemisorption; Adsorption; Dendrimer; Pseudo-Second-order kinetic model; Harkins-Jura; Baudu

Introduction
The removal of heavy metal ions from industrial effluents is among the worst environmental challenges facing the world today. Heavy metals are chemical elements with a specific gravity that is at least 5 times the specific gravity of water. They are normally challenging environmental pollutants, with renowned toxic effects on living systems [1].

Heavy metals are really hazardous because they are persistent and could remain in the environment long after the original source of pollution is done away with [2].

The mobility of heavy metal ions in ground-water systems is slowed down by reactions that cause metal ions to adsorb or precipitate making them have a propensity to be associated with the solid phase and this usually prevents them from dissolving. While various heavy metal ions undergo similar reactions in a number of aspects, the extent and character of these reactions vary under particular conditions. The uptake of heavy metal ions by plants and subsequent accumulation along the food chain remains a potential threat to human health [3].

Cadmium is a heavy metal with atomic weight 112.4 g/mol it occurs naturally in zinc and lead ores, sedimentary rocks and phosphate fertilizers too [4,5]. Cadmium has been used in the production of alloys, pigments and batteries thus it poses considerable environmental and occupational health concern [6].

Some major sources of widespread cadmium distribution into the environment are: industrial applications, use of phosphate fertilizers in agriculture and sewage sludge. It can also be dispersed via natural emissions and bioaccumulation processes that take place in some plants, mammals and even filter feeder organisms like crustaceans and mussels [7]. Other prominent routes for human exposure to cadmium are inhalation of cigarette smoke and ingestion of cadmium infested food and water. Studies have shown that cadmium is very toxic to humans even at very low concentrations because it causes damage to the human body cells through the generation of Reactive Oxygen Species (ROS) that cause single-strand DNA damage which interrupts the synthesis of nucleic acids and proteins [8-10]. Studies have also shown that heightened cadmium intake can cause gastrointestinal tract erosion, pulmonary, hepatic/renal injury and obstructive lung disease [11].

Silica gel, a non-toxic and chemically inert compound composed of amorphous silicon dioxide is used in water purification studies amongst other uses due to its high surface area (800 square meters per gram) arising from the micro-porous structures of interlocking cavities [12,13].

Methods for treating industrial wastewater containing heavy metals often involve processes of toxicity reduction in order to meet technology-based treatment [14]. Some of such heavy metal treatment techniques include chemical precipitation, ion exchange, coagulation/flocculation, ultra-filtration, electrochemical treatment, electrodialysis and adsorption. Adsorption is a well-known, uncomplicated, reasonably priced and proficient means of eradicating heavy metal ions from waste-water using a range of adsorbents such as Bentonite, Fly Ash, Layered Double Hydroxides, Metal Organic Frameworks and many other carbon-based materials [15].

In this study Generation-5 Polyamidoamine dendrimer was immobilized on chromatography grade silica gel to produce a functionalized dendrimer-silica composite (G-5-PAMAM-SGA). The composite was characterized using Fourier Transform-Infra Red spectroscopy, Brunauer Emmett and Teller surface area analysis, Thermogravimetric Analysis, Zeta Potential, and Scanning Electron Microscopy. Adsorption properties of the synthesized composite were studied by batch adsorption method using Cd (II) as adsorbate. The sorption process was analyzed using 6 different isotherm models (Langmuir, Freundlich, Harkins-Jura, Sips, Jossens and Baudu).

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functionalized with Succinic anhydride and subsequently immobilized on chromatographic grade silica gel to produce a dendrimer-silica gel composite that was characterized in order to reveal its physicochemical properties. The prepared adsorbent was then tested for its adsorption of Cadmium (II) ions in aqueous solution under optimized reaction conditions of contact time, pH, initial concentration and temperature.

Materials and Methods

Synthesis of G-3 PAMAM functionalized silica

All chemicals used for this study were of Analytical grade. G-5 polyamidoamine (PAMAM) dendrimers with ethylenediamine core, succinic anhydride, cadmium nitrate salt (3-aminopropyl) triethoxysilane (APTES) and chromatographic grade silica gel (particle size-240-425 mesh, pore size-15 nm, pH-7, pore volume-1.15 cm3/g) were obtained from Sigma Aldrich, South Africa. While N-(3-dimethylaminopropyl)-N'-ethylcarbodiimide hydrochloride was procured from Thermo Fisher scientific, Belgium.

Grafting APTES on silica

APTES (3-aminopropyl) triethoxysilane) was grafted on silica gel in accordance with a procedure used by Acres et al. [16]. One of the precursor materials silica gel (30 g) was oven dried at 130°C for two hours; by this silica gel crystals were successfully activated. The activated silica gel was then refluxed at 115°C for six hours in a 10% APTES/anhydrous toluene mixture. The resultant product obtained is the amino functionalized silica gel which was subsequently separated from the solution by centrifugation at 4000 rpm for 10 min. The amino functionalized silica gel was washed using water and ethanol alternatively in order to remove excess reagents. The very last wash was done with ethanol after which the product was oven dried at 110°C for one hour.

Functionalization of G-5-PAMAM dendrimer

Another precursor material, Succinic acid terminated generation-5 PAMAM dendrimers was prepared using the method of Shi et al. [17]. Thus, succinic acid terminated generation-5 PAMAM dendrimer was prepared by dissolving about 3.7 mL of G-5-PAMAM dendrimer and 3.1 g succinic anhydride in separate 50 mL volumes of Dimethyl Sulfoxide (DMSO). Both solutions were then transferred into a round bottom flask and refluxed at 80°C for 12 h and dialyzed with deionized water over 3 d (the deionized water was replaced every 6 h). The succinic acid terminated generation-5 PAMAM was withdrawn from the dialysis setup by means of a micropipette and stored for further use.

Coupling functionalized dendrimer unto APTES grafted silica

The final product G-5-PAMAM was prepared using the method of Jiang et al. [18], in which the two precursor materials; silica gel grafted with APTES (20 g) and succinic acid terminated generation-5 PAMAM dendrimer (20 mL) were added into 250 mL round bottom flask containing 75 mL methanol. A coupling agent N-(3-dimethylaminopropyl)-N'-ethylcarbodiimide hydrochloride (EDC) (5 mg) was added to the flask and the entire mixture was refluxed at 90°C for twelve hours [19]. The final product was separated from excess reagent by centrifugation at 4000 rpm for about 10 min, washed three times with ethanol and oven dried at 110°C for one hour. The final product is the G-5-PAMAM functionalized silica composite (G-5-PAMAM-SGA).

Characterization of the adsorbent

The G-5-PAMAM-SGA and pristine silica were characterized using Fourier Transform Infrared (FTIR) spectrometer in order to determine the associated functional groups using (Spectrum Two, Perkin Elmer Instruments, USA), determination of surface area and porosity was done using Micromeritics TRISTAR II 3020 analyzer (Micromeritics Instrument Corporation, USA). A Zeiss Auriga Field Emission Scanning Electron Microscope (SEM) was used for determination of the adsorbent's surface morphology while thermal stabilities of both materials were determined using thermo-gravimetric analyzer (Perkin-Elmer TGA 4000 by Perkin Elmer Instruments, USA).

Adsorption study

Cadmium nitrate salt was used in the preparation of the stock solution (1000 g/L), after which working solutions were prepared from the stock by serial dilution. Batch adsorption of Cd (II) ions was carried out using G-5-PAMAM-SGA as adsorbent in order to determine the effects of contact time, concentration, pH and temperature. The adsorption procedure was performed as follows; 20 mL of absorbate solution (specific concentrations) was added to 20 mg of adsorbent material. The adsorbent/adsorbate mixture was then agitated by means of an orbital shaker operating at 200 rpm.

Desorption study

Desorption study was done using 20 mg of previously used adsorbent. Cd (II) ions that were initially adsorbed were desorbed by shaking in 20 mL 0.5 M nitric acid at 220 rpm for duration of 10 min. The plastic centrifuge bottles were removed from the shaker and placed on a centrifuging machine operated at 3500 rpm for 10 min. The amount of Cd (II) ion left in the solution was determined using Atomic Adsorption Spectrometry. The adsorbent was washed twice with dilute nitric acid (0.5 M) and lastly with ultra-pure water before the second round of re-use test (second cycle). The re-use test was carried out for three cycles.

Data management

The adsorption capacity (q) and removal capacity (R%) of Cd(II) ions by G-PAMAM-SGA were calculated using the following equations:

\[ q_e = \frac{(C_0 - C_e)V}{M} \]  \hspace{1cm} (1)

\[ R(\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \]  \hspace{1cm} (2)

Where \( C_0 \) and \( C_e \) (mg/L) are the initial and equilibrium concentrations of Cd(II) ions in solution, \( V \) is the volume of adsorbate solution used (mL) while \( M \) is the mass (g) of adsorbent used for the experiment.

Three kinetics isotherm models Pseudo-First Order (PFO) [20] (Eqn 3), Pseudo-Second Order (PSO) (Eqn 4) kinetics models, and the Weber-Morris [21] intraparticle diffusion (IPD) (Eqn 5) were used in describing the effect of time data.

\[ q_t = q_e (1 - e^{-k_1t}) \]  \hspace{1cm} (3)

\[ q_t = \frac{q_e^2k_2t}{1 + q_0k_2t} \]  \hspace{1cm} (4)

\[ q_e = k_{IPD} t^{1/2} + C \]  \hspace{1cm} (5)

The symbols \( q_t \) and \( q_e \) are the amounts of Cd(II) adsorbed (mg/g) on the adsorbent (G-5-PAMAM-SGA) at equilibrium and time \( t \), respectively; and \( k_1 \) (min\(^{-1}\)), \( k_2 \) (g/g/min) and \( k_{IPD} \) (g/min\(^{1/2}\)) are the rate constants of the PFO, PSO and IPD, respectively; while \( C \) (mg/g)
is the amount of Cd(II) adsorbed on the adsorbent surfaces. Model parameters were generated from the KryPlot software.

Equilibrium data at 294 K were calculated using Langmuir (Eqn 4), Freundlich (Eqn 5), Harkins-Jura (Eqn 6), Jossens (Eqn 7), Sips (Eqn 8) and Baudu (Eqn 9). Langmuir, Freundlich and Harkins-Jura equations can be written in the following linear forms [22].

\[ C_e = \frac{1}{q_m} + \frac{C_e}{K_L q_m} \]  
\[ C_e = q/\text{Hexp}(Fq_e) \]  
\[ 1/n = \text{Adsorption intensity, it also indicates the relative distribution of the energy and the heterogeneity of the adsorbent sites.} \]

Where B and A are Harkins-Jura constants that can be obtained from plotting \( 1/q_{\text{ce}} \) vs \( \log C_e \) and

\[ \ln(1-\theta) = \ln S^* + \frac{\Delta S^*}{RT} \]  
\[ \text{Where } \theta \text{ is the degree of surface coverage.} \]

Results and Discussion

Physical and chemical characterizations

Results of the physical and chemical characterization of G-5-PAMAM-SGA confirmed the successful synthesis of the composite (G-5-PAMAM-SGA) are shown in Figure 1a, the point of zero charge (pHpzc) was 2.9. Thus, the surface of G-5-PAMAM-SGA becomes predominantly negative above this pH value. The \( N_{\text{a}} \) adsorption/desorption isotherm (not shown) were the classical type IV isotherm, which is an indication for a mesoporous material; the BET surface area was 4.0 m²g⁻¹ with a relatively large pore width of 22.4 nm which is higher than that for many reported silica-based adsorbents [23]. This large pore width has more space for large contaminant molecules to be removed from solution via pore filling [24]. The FTIR spectra of the pristine Silica gel and G-5-PAMAM-SGA are shown in Figure 1b. The peaks observed at 1060 and 1800 cm⁻¹ are characteristic peaks for silanol groups [23]. While the peak at 1630 cm⁻¹ is attributable to the newly introduced amide group.

Adsorption rate and Kinetics of Cd(II) uptake by G-5-PAMAM-SGA

The TGA spectra (Figure 1c) showed two major thermal transitions for the pristine silica gel and G-5-PAMAM-SGA as temperature was gradually increased from 40 to 900°C. The initial transition was observed below 150°C for both materials, this was ascribed to loss of physisorbed water molecules within the layer bed of the basic silica material [23]. The higher weight loss exhibited by G-5-PAMAM-SGA at lower temperatures confirms the presence of APTES and PAMAM dendrimers groups which were more labile than the backbone silica material. Thus, the weight loss at this second thermal transition could be attributed to the endothermic decompositions of surface hydroxyl groups on the pristine silica gel and G-5-PAMAM-SGA as well as the APTES and PAMAM groups. The pre and post-adsorption G-5-PAMAM-SGA SEM images are shown in Figures 1d-1e. The Figure 1e showed shiny agglomerated surfaces due to the presence of the adsorbed Cd(II) cations.
be attributed to pore filling [25]. As such the C value given in Table 1 reveals that approximately 82.66% was due to surface adsorption and 17.34% due to pore filling.

**Effect of pH on Cd(II) uptake by G-5-PAMAM-SGA**

Scientific investigations have shown that pH affects the charge density around adsorbate and adsorbent molecules [26]. Following the aforementioned the uptake of Cd(II) by G-5-PAMAM-SGA was investigated as pH was varied (Figure 3). The observed trend clarifies the effect of pH with respect to the point of zero charge of the adsorbent (2.9). Below the point of zero charge (2.9) the adsorbent becomes predominantly positively charged because of protonation of its surface-active functional groups, thus adsorption at this stage could be mainly due to pore filling. But with increase in pH value the surface of the adsorbent become negatively charged due to deprotonation and this facilitates electrostatic interactions between Cd(II) ions and the adsorbent. Therefore, higher adsorption values were observed as shown in Figure 3.

**Effect of concentration and temperature on the uptake of Cd(II) ions by G-5-PAMAM-SGA**

The trend showed an increase in Cd(II) adsorption by G-5-PAMAM-SGA as initial concentration (Co) increased from 75 through 175 mg/L. The drift was also observed at 20, 30 and 40°C respectively (Figure 4). This trend may be attributed to the activities of Cd(II) ions between the surface and the internal cavities of the adsorbent. At equilibrium, migration of Cd(II) ions between adsorbent surfaces and pores may be equal for specific concentration values (Co), therefore, movement of the ions across both boundaries would have been retarded. Nevertheless, increasing the concentration of the Cd(II) ions improved the migration of adsorbate molecules across both boundaries thus, there is an observed increase in adsorption [27].

**Table 1: Kinetic parameters for the adsorption of Cd (II) by G-5-PAMAM-SGA.**

<table>
<thead>
<tr>
<th>Model</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>PFO</td>
<td>q_e (mg g⁻¹)=7.360, K₁ (min⁻¹)=2.1 × 10⁻⁴, R²=0.764</td>
</tr>
<tr>
<td>PSO</td>
<td>q_e (mg/g)=5.128, K₂ (g mg⁻¹ min⁻¹)=2.535, R²=0.999</td>
</tr>
<tr>
<td>IPD</td>
<td>C (mg g⁻¹)=86.934, K_id (g⁻¹ min⁻¹)=0.82799, R² = 0.5335</td>
</tr>
<tr>
<td>PPA</td>
<td>17.34</td>
</tr>
<tr>
<td>PSA</td>
<td>82.66</td>
</tr>
<tr>
<td>PPA: Proposed Pore Adsorption as predicted by IPD</td>
<td></td>
</tr>
<tr>
<td>PSA: Proposed Surface Adsorption as predicted by IPD</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1: (a) Point of zero charge plot for G-5-PAMAM-SGA, (b) FTIR spectra of pristine silica gel and G-5-PAMAM-SGA, (c) TGA spectra of pristine silica gel and G-5-PAMAM-SGA, SEM micrograph of G-5-PAMAM-SGA, (d) Before Cd(II) adsorption and (e) After Cd(II) adsorption.

Figure 2: Adsorption trend for Cd(II) as time increased.
Equilibrium data at 293K were assessed using Langmuir, Freundlich, Harkins-Jura, Jossens, Sips and Baudu isotherm models, the isotherm parameters are shown in Table 2. Comparison of parameters of the models showed that experimental data fitted the Jossens model more than the other models. Jossens model (r²=0.981), Baudu (r²=0.973), Harkins-Jura (r²=0.9607), Sips (r²=0.953), Freundlich (r²=0.952), and Langmuir (r²=0.935). The good fitting of Jossens model with experimental data implies that the adsorbent has heterogeneous surface. Similarly, the high correlation coefficient obtained from Harkins-Jura model indicates that the adsorbent is heteroporous. Freundlich isotherm also showed a good fit to experimental data with n>1, indicating chemisorption. Freundlich isotherm also showed good adsorption capacity (K_f=58.57) which suggests a multi-layered adsorption on a heterogeneous surface of unequal energy which is consistent with a previous report [28].

Increasing the ambient experimental temperature from 20 through 40°C showed that temperature had a positive effect on the adsorption of Cd(II) ions on G-5-PAMAM-SGA; therefore, increasing the temperature, increases Cd(II) adsorption. This trend suggests an endothermic process for Cd(II) ions adsorption on the adsorbent. In order to determine this, the adsorption process was further examined using thermodynamic parameters (ΔH°, ΔS° and ΔG°) which were estimated from the experimental equilibrium data obtained at the various temperatures investigated and the parameters are shown in Table 3.

The ΔG° values for all temperatures studied were negative, this suggested spontaneous and feasible Cd(II) adsorption processes. The value of enthalpy change (ΔH°) was positive and this confirmed the supposition that the adsorption process was endothermic. In an endothermic process, any increase in external energy favors the forward process; consequently, Cd(II) adsorption improved with increase in solution temperature [29]. The positive value of the ΔS° is an indication of increased randomness of the Cd(II) ions in solution as the adsorption process progressed towards equilibrium. The calculated value for sticking probability (S') was approximately equal to 1, this signifies an adsorption mechanism having a combination of chemisorption and physisorption [30]. The low value of activation energy (Ea) is an indication of a diffusion-controlled sorption process [31-39].

**Reusability and comparison with reported adsorbents**

Desorption studies was carried out in order to appraise the economic prospects in the use of G-5-PAMAM-SGA as an adsorbent for the removal of Cd(II) ions from aqueous solutions. Results of three cycles reusability test using previously used G-5-PAMAM-SGA as adsorbent for Cd(II) removal is illustrated with Figure 5 it can be

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**Table 2:** Adsorption isotherm models parameters for Cd (II).

<table>
<thead>
<tr>
<th>Model</th>
<th>q_max (mg/g)</th>
<th>b (L/mg)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Langmuir</td>
<td>20.33</td>
<td>7.49</td>
<td>0.9352</td>
</tr>
<tr>
<td>Freundlich</td>
<td>58.57</td>
<td>0.2199</td>
<td>0.9525</td>
</tr>
<tr>
<td>Harkins-Jura</td>
<td>5000</td>
<td>195.191</td>
<td>0.9607</td>
</tr>
<tr>
<td>Jossens</td>
<td>1.776</td>
<td>195.191</td>
<td>0.9805</td>
</tr>
<tr>
<td>Sips</td>
<td>5000</td>
<td>195.191</td>
<td>0.9607</td>
</tr>
<tr>
<td>Baudu</td>
<td>21.792</td>
<td>8.2716</td>
<td>0.9726</td>
</tr>
</tbody>
</table>

**Table 3:** Thermodynamic parameters of G-5-PAMAM-SGA for Cd (II) adsorption.

<table>
<thead>
<tr>
<th>Thermodynamics</th>
<th>Parameters</th>
<th>Cd (II)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ΔH° (kJ mol⁻¹)</td>
<td>10.474</td>
<td>11.399</td>
</tr>
<tr>
<td>ΔS° (J mol⁻¹ K⁻¹)</td>
<td>293 K</td>
<td>-3329.9</td>
</tr>
<tr>
<td>ΔG° (kJ mol⁻¹)</td>
<td>303 K</td>
<td>-3443.27</td>
</tr>
<tr>
<td>Ea (kJ mol⁻¹)</td>
<td>3.3 × 10⁻²</td>
<td>313 K</td>
</tr>
<tr>
<td>S'</td>
<td>0.9999</td>
<td>0.6502</td>
</tr>
</tbody>
</table>

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Figure 3: Effect of pH on the uptake of Cd(I).

Figure 4: Effect of initial concentration and operating temperature on the uptake of Cd(II).
deduced that the third cycle was slightly lower than the second cycle by approximately 35%.

The high adsorption exhibited in the first and second cycles could be due to availability of empty adsorption sites on the adsorbent. Meanwhile, desorption of Cd(II) ions during the first and second cycles was simple because the desorbing solvent could reach the surface adsorbed adsorbates easily, but adsorbates molecules that were initially adsorbed into adsorbent pores and crevices were not accessible at the third cycle of desorption. This led to a reduction in the removal capacity of the adsorbent by 35%.

The adsorption of Cd(II) ions by G-5-PAMA-SGA was compared with that of other adsorbents as shown in Table 4. The comparison revealed that G-5-PAMAM-SGA performed better that a number of adsorbents in the removal of Cd(II) ions. This serves as an indicator for the potentials of G-5-PAMAM-SGA as an effective adsorbent for the removal of Cd(II) ions from wastewater.

Conclusion

In this study G-5-PAMAM-Silica gel composite (G-5-PAMAM-SGA) was synthesized, characterized and used for the removal of Cd(II) ions from aqueous solution. Characterization results showed peaks of associated functional groups, larger pore size and slightly lower thermal stability when compared with unmodified silica gel. All proving a successful synthesis of G-5-PAMAM-SGA. SEM images revealed the presence of agglomerated Cd(II) ions on G-5-PAMAM-SGA. Equilibrium results showed that equilibrium was reached after 180 min, optimum pH=6 and q =123.4 mg/g which is better than that of several adsorbents in literature. Thermodynamic studies indicated endothermic, spontaneous and feasible adsorption process. The adsorption modelling parameters indicated multi-layered sorption onto heterogeneous/heteroporous adsorbent. Two processes were implicated in the sorption of Cd(II) ions by G-5-PAMAM-SGA namely; adsorptive pore filling of empty mesopores and electrostatic interactions between negatively charged surface groups and Cd(II) ions in solution.

Pseudo-second-order kinetic model described the adsorption process accurately thus, it was mainly controlled by chemisorption. Jossens, Harkins-Jura, and Freundlich models were the most reliable isotherm models for describing the sorption process as evidenced by high correlation coefficients. G-5-PAMAM-SGA exhibited good reusability after three cycles of desorption. These results imply that G-5-PAMAM-SGA could be efficient in the removal of Cd(II) ions from aqueous phases most especially during waste-water treatment procedures.

References