Remediation of Boating and Jetty Pollutants from the Okavango Delta Surface Water Employing Valorized *Tilapia ruweti* Waste Material

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Authors’ contributions

All the authors contributed significantly to this work. Together, the authors designed the study, performed the statistical analysis, wrote the protocol and drafted the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

**Aims:** To employ valorized waste materials from *Tilapia ruweti* scales for removal of heavy metals from areas with major boating activities and high numbers of jetties at the Okavango delta, Botswana.

**Study Design:** Biowaste materials were identified, valorized and then optimized for adsorption and removal of boating and jetty pollutants from profiled Okavango Delta surface water samples.

**Place and Duration of Study:** Okavango Delta, Maun and Botswana International University of Science and Technology, Palapye, Botswana between March 2017 and September 2018.

**Methodology:** *Tilapia ruweti* scales were collected from Sehithwa, Maun while water samples were collected from areas with jetties and major boating activities at Shakawe, Mboma Island and Xakanaxa. The concentration of Zn, Cu, Ni, Co, Pb and Cd at all the sampling sites were accurately determined using micro-plasma atomic emission spectroscopy (MPAES). The fish scales were pulverized and valorized by subjecting them to 12.7% vinegar. Minitab 14 software was used as a modeling tool to provide multivariate optimized parameters that affect sorption studies that included initial ion concentration, sorbent dose, contact time and solution pH. The valorized waste materials were then utilized for removal of the selected heavy metals.
Results: The optimized adsorption parameters that included contact time, solution pH, sorbent dose and initial ion concentration were ≤ 88.63 min, ≤ 8.75, ≤ 84.29 mg and ≤ 28.44 mg/L respectively. The valorized *Tilapia ruweti* waste displayed high removal efficiencies toward removing the selected ions from the Okavango Delta surface water samples up to 94.21% with %RSD < 2 for n = 3 (triplicate).

Conclusion: The valorized *Tilapia ruweti* scales were recommended as a cheap, simple and an effective method for remediation of boating pollution at the Okavango delta and other recreational areas.

Keywords: Boating pollution; Okavango delta; heavy metals; jetties; valorized; *Tilapia ruweti*.

1. INTRODUCTION

Okavango Delta (OR) located on the north-west of Botswana is one of the most famous destinations owing to its pristine waters, flora and fauna. Due to its rich wildlife diversity and scenic beauty, this region has attracted tourist from all parts of the world, making tourism the second Gross domestic product contributor to Botswana’s economy [1,2]. Over the last few years, with an increase in leisure time available to a large number of the working population, the opportunities to pursue outdoor recreational activities have increased. Most tourism development adds pressure on the environmental resources upon which it is based, compromising the future prospects of the local population and, indeed, the expectations of tourists themselves [3,4]. Tourism contains the seeds of its own obliteration, tourism can kill tourism, destroying the very environmental attractions which visitors come to a location to experience [5]. The growing use of rivers for recreational purposes has led to concerns about increasing anthropological pressures on freshwater environments. In this case, use of motorized recreational and commercial boats on the Okavango Delta water bodies has risen significantly, and it is likely that this trend will continue. During baseline studies, it was established that jetties which are walkway structures that projects from the land out into the water [6] and boats are sources of pollution at the river banks. The walkway helps boat owners and tourist to access and board the boats that are at the center of an enclosed waterbody. Boats and jetties spend a large proportion of their working life partly submerged in water. As with all objects subjected to long periods of time in the water, they become prone to corrosion which inhabit the aquatic environment [6-8]. Fig. 1. shows pictures of a typical jetty and boats used at the Okavango Delta.

![Fig. 1. Pictures of a jetty (A) and boats (B) used at the Okavango Delta](image)

Both jetties and boats are source of heavy metals as they are coated with metal-based antifouling paints that leach anti-fouling agents such as cadmium, nickel, cobalt and lead into the water bodies [9,10]. In addition the jetties are constructed using materials made of metals including zinc, copper, and iron [11]. These pollutants have been proven to be highly toxic, even in minute amounts, and tend to accumulate in organisms, often producing mutagenic effects [12,13]. Most studies have come to the conclusion that recreational boating activities can create a number of problems for aquatic environments and their surrounding areas [14-16]. Some of the aquatic animals drink water or rest at the river banks which is a home of pollution due to the aforementioned activities. Furthermore, the riparian communities not only depend on the Okavango delta as a source of employment but also as source of water for domestic and agricultural purposes [17,18].
In Botswana, where this study was conducted; to the best of our knowledge, little or no effort has been directed towards studying the effects of boating pollution on the environment. There is a need to bring awareness to relevant stakeholders on the effect of boating activities and inform boat owners on how to remediate pollution that originate from such activities. Consequently, prevent transportation of the pollutants to the rest of the Okavango Delta. Recently there has been an increasing trend to evaluate some indigenous cheaper biological materials for remediation of polluted water. The cost implications are more serious to the developing countries. Therefore, in this paper, affordable and more environmentally friendly materials for removing heavy metals from contaminated water were sought. Valorized waste materials from *Tilapia ruweti* scales were employed for removal of heavy metals from area with major boating activities and high numbers of jetties at the Okavango Delta, Botswana.

## 2. METHODOLOGY

### 2.1 Materials and Instrumentations

*Tilapia ruweti* waste were sampled at Lake Ngami in Sehithwa, Maun, Botswana. HCl (37%) and HNO3 (67%) analytical reagents were obtained from Sigma Aldrich (Missouri, United States), NaOH (97%) pellets and 1000 ppm standard solutions of Cd, Cu, Zn, Ni, Co and Pb were purchased from Rochelle Chemicals (Johannesburg, South Africa). Filtering procedures were conducted using 0.45 μm Whatman filter papers (Hardened Ashless Circles 45 mm) obtained from Sigma-Aldrich (Johannesburg, South Africa). White Vinegar used to modify the waste materials, was obtained from SPAR (Palapye, Botswana). Extraction of waste materials from *Tilapia ruweti* scales were sampled at Lake Ngami in Sehithwa near Maun, Botswana were treated and characterized using a Mars6 One Touch Microwave Assisted Extractor/Digester obtained from CEM Microwave Technology Ltd, (North Carolina, USA). The fish scales were characterized using JSM 1700 SEM coupled with EDX from JEOL Ltd (California, USA), PerkinElmer Fourier transform infrared (FTIR) spectroscopy obtained from Chemetrix (Pty) Ltd, (Johannesburg, South Africa) and a powder D8 Advanced Powder X-Ray Diffractometer (XRD) from Bruker Inc. (Karlsruhe, Germany). Agilent 4100 microwave-plasma atomic emission spectroscopy (MP – AES) purchased from Chemetrix (Pty) Ltd, (Johannesburg, South Africa) was used to determine the concentrations of the heavy metals.

### 2.2 Sampling and Sample Handling

Sampling design employed at the Okavango delta targeted areas with motorized boating activities and areas with jetties. Water samples were collected from Shakawe, Mboma Island, Xakanaxa and Boro Island (only wooden boats are used and they do not use jetties). Fig. 2 shows the specific areas of sampling sites. The pH, dissolved oxygen and conductivity measurements of the surface were conducted on site. The pH of water was reported to be relatively neutral, ranging from 7.08 to 7.64, which was within the acceptable minimum limits of 6.5 to 8.5 for potable water [18]. Mean electrical conductivities of delta’s water increased from 46.3 μScm⁻¹ upstream (Shakawe) to 107.3 μScm⁻¹ in Matlapeng. The values were below recommended 1000 μScm⁻¹ conductivity limits for surface water [19]. All the sampling sites had mean dissolved oxygen values ranging from 4.1 to 5.9 mgL⁻¹, which were higher than 2.4 mgL⁻¹ required by aquatic life [20]. The water samples were sampled using grab sampling method and preserved in plastic bottles at pH <2 at < 4°C whereas the sediments were sampled at the banks of the river, sun dried for 48 hours and stored in aluminum foil in a freezer (<0°C) till further analysis.

### 2.3 *Tilapia ruweti* Scales Remain Valorization and Characterization

The fish scales waste from *Tilapia ruweti* (Tr) sampled at Lake Ngami in Sehithwa near Maun, Botswana were treated and characterized following an adapted method by Stevens and Batlokwa [21]. The scales were sun dried for 2 days and pulverized using a Fritsch pulverisette 5 pulvizer obtained from Fritsch (Berlin, Germany), then sieved to 63 – 200-micron mesh size. After screening, they were washed several times with deionized water to remove color and dust. The waste materials were then valorized by treating with white vinegar to remove inorganic pollutants. The valorized fish scales were then dried in an oven at 65±5°C for 6 hours and characterized using FTIR, XRD and SEM-EDX.

The FTIR data were collected at 2.0 cm⁻¹ resolution, and each spectrum was as a result of 256 scans which were recorded in the wavelength range 500-4000 cm⁻¹ on a Nicolet iS10 Thermo Scientific FTIR. The XRD was operated with Cu Kα emission (λ = 1.54105Å, 40 kV, 40 mA per sec) and with high efficiency linear detector of Lynx Eye type. The crystallite size of the sample was calculated by Deby-Scherrer
method. The scanning mode used was coupled with 2θ/θ on the scanning range 10°-120° values. The Tr scales were carbon coated using an E6700 Polaron range high vacuum pressure sputter coater (Quorum Technologies, UK). The carbon coated Tr were then taken for SEM-EDX analysis, which was operated under high vacuum and beam acceleration voltage of 10.0 kV (the recommended operating voltage for organic material samples). The obtained results were used to determine the morphology and elemental composition of the Tr scales waste.

2.4 Concentrations of Selected Heavy Metals in *Tilapia ruweti* Scales

Aqua regia solution of HCl: HNO₃ at a ratio of 3:1 v/v in a MARS6 microwave assisted digester was used for extraction of heavy metals from the Tr scales. 1 g of the Tr scales powder was weighed and placed in 100 mL TFM sample vessel. 20 mL of the aqua regia solution was added and the mixture was digested at 600 psi, 100°C and 1200 W. The ramp time was set at 20.0 min with a hold time of 10.0 min. The subsequent volume was filtered using a whatman No. 1 filter paper and transferred into 50 mL volumetric flasks. The sample was then diluted with deionized water up to the mark. The concentrations of the metals in the samples were then determined using MP-AES.

2.5 Adsorption Studies for Removal of Selected Heavy Metals Employing *Tilapia ruweti* Scales

Standard solutions of Cd, Zn, Ni, Co, Pb and Cu (100 mg/L) were prepared from 1000 ppm stock solution of each of the metals. The valorized Tr scales powder of ≤ 63 μm particle size were used. Batch experiments using multivariate optimization methodologies were conducted. Minitab Release 14 statistical software (Minitab Inc., USA) was employed to model the optimization experiments. At; the experiments were conducted in triplicates to evaluate the adequacy of the method and lack-of-fit.

2.5.1 Optimization of adsorptive parameters

Four factors that affect adsorption including contact time, pH, sorbent dosage, and initial concentration were optimized. Firstly, a two-level fractional factorial design was conducted for screening purposes. The factors that were significant towards the experimental output were identified. Afterwards, a face centered central composite design was then conducted to determine the optimum conditions for each factor, resulting to a maximized response of the experiments. During the screening process, the experimental conditions in Table 1 were adapted [21]. The resulting filtered solution was transferred into a 50 mL volumetric flask and the sample diluted with deionized water up to the mark. The concentration of the metals in the sample solution was then determined using MP-AES.

After identifying the significant factors from the screening phase, optimization phase (CCD) was then used as the experimental matrix for the response surface method. The resulting filtered solution was transferred into a 50 mL volumetric flask and the sample was diluted with deionized water up to the mark. The concentration of the metals in the sample solution was then determined using MP-AES.

Table 1. Two-level fractional factorial design for the optimization of the valorized TR scales

<table>
<thead>
<tr>
<th>Factors</th>
<th>Adsorption parameters</th>
<th>Low level (mg)</th>
<th>High level (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Adsorbent dosage</td>
<td>25</td>
<td>1000</td>
</tr>
<tr>
<td>B</td>
<td>pH</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>C</td>
<td>Contact time (min)</td>
<td>15</td>
<td>90</td>
</tr>
<tr>
<td>D</td>
<td>Metal Concentration</td>
<td>0</td>
<td>50</td>
</tr>
</tbody>
</table>

2.6 Application of Optimal Valorized *Tilapia ruweti* Scales

Valorized Tr scales waste remains (VTr WR) were used to remove selected metals from water samples collected from areas with boating pollution at the Okavango Delta, Botswana. The VTr WR were used for Zn, Cd, Pb, Ni, Co and Cu removal in the water samples. 50 mL of Okavango water sample were placed in a 100 mL flask and optimized conditions were applied to the water samples. The mixtures were shaken at 200 rpm for the optimized time, after which they were filtered. The resulting filtrate was transferred into a 50 mL volumetric flask and then diluted up to the mark using deionized water. Calibration curves were determined using 5.0 to 50.0 mg/L metal standards solutions prepared from the 1000 mg/L standard solutions. The concentration of the metals in the water samples after adsorption were determined with MP-AES. Mean values for the percentage removal and the relative standard deviations (RSD) were calculated as per Equation 1.
3. RESULTS AND DISCUSSION

3.1 Valorization

Following the procedures described in section 2.3, to valorize (rejuvenate) the collected fish scales, pulverization and sieving of the collected waste material was the first procedure that was conducted. The procedure resulted in off-white powdery material (see Fig. 3, a camera picture of the fish scale powder) of smaller particle sizes of < 63 µm as estimated from the sieve’s mesh sizes.

Further elucidation on the particle size, shape and morphology of the particles was achieved by employing the SEM which revealed morphologically rough-surface (see Fig. 4 B) particles of cylindrical geometry and particle sizes of about 10 µm as evidenced in the SEM micrographs in Fig. 4 A & B.

The cylindrical geometry (see Fig. 4B) and smaller particle sizes are known to be good characteristics for effective sorbents as they result in high surface area to volume ratio as well as a larger exposed surface area of the adsorbent to the targeted species [22]. The large Tr scale remains that were originally collected (before pulverization and sieving) were cumbersome to handle, thus pulverization of the waste remains achieved preliminary valorization of the waste materials. To valorize a waste material is to put to use or to improve (rejuvenate) its usage after it had being deemed useless and regarded as waste. Prior to further valorization of the pulverized material, it was subjected to microwave digestion as described in Section 2.4. After analysis employing MP-AES, it was discovered that the pulverized material contained lead, nickel, copper, zinc and iron ions but below maximum acceptable levels set out by World Health Organization (WHO) [23,12,24]. Since the Tr scale waste materials that were pulverized could be better handled in powder form, they were thereafter treated with vinegar to further valorize them.

Vinegar treatment was performed to further valorize the waste materials in order to expose the surface functional groups needed for adsorption that could have been embedded in
the structures of the waste remains. The volume of the 12.7% vinegar needed to effectively wash the waste remains such that maximum adsorption or removal efficiency of the selected ions was achieved and optimized as described in Section 2.3. The point at which plots of percentage adsorption or removal efficiency versus volume of acid plateaued, marked the optimized volume of vinegar that was needed to wash the pulverized waste remains for maximum valorization as represented by high adsorption percentage efficiencies in Fig. 5.

After valorization of the Tr scales, XRD was used to determine the effect of valorizing the waste material. The VTrWR and the non-VTrWR were analyzed for the presence of metal ions residuals or compounds. As evidenced in Figs. 6 and 7 (VTrWR and the non-VTrWR respectively), the diffractogram of the VTrWR showed no evidence of the presence of metallic compounds or residues, while, the diffractogram of the non-VTrWR, revealed the presence of metallic compounds or residues. This revelation further supported the effectiveness of the use of vinegar for valorization of the waste materials.

3.2 Fourier Transform Infrared Spectroscopy (FT-IR)

The functional groups on the VTrWR that are responsible for the adsorption of the targeted ions were determined using Fourier transform infrared spectrometer (FTIR). The FTIR spectra of the VTrWR showed stronger vibrational bands that were shifted when compared to the spectra of their non-VTrWR counterparts (see Fig. 8). The shifted, strong bands were attributed to the freed, non-bonded functional groups on the VTrWR that were exposed by treating the pulverized waste materials with vinegar. Usually, the functional groups occur naturally coordinated (bonded) to various ions as supported by the XRD diffractogram (see Fig. 7).

The most prominent bands found in the spectra (Fig. 8) were the carboxyl groups located around 1398 cm⁻¹, primary amines around 733 cm⁻¹, amide groups around 2926 cm⁻¹, alkanes at 887 cm⁻¹, and hydroxyl groups around 1646 and 1746 cm⁻¹ which are known to form coordinate bonds with ions through their lone pairs as well as hydrogen bonding for the ones with hydrogen atoms in their structures [25,26].

3.3 Optimization of Adsorptive Parameters

In order to optimize the practical experiments that were to be carried out with the VTrWR, Two-
Fraction Factorial Design and Central Composite Design (CCD) in Minitab statistical software, was used to model the experimental parameters (discussed in Section 2.5.1) that influence binding/adsorption.

Fig. 9 illustrates the interaction between adsorption parameters, the standardized effects and their importance on the adsorption of the metals onto the valorized Tr scales. In each of the chart, the reference line (at 2.31), which was obtained based on error terms (in cases of error term) or Lenth’s pseudo-standard error (in cases of no error term), separates the significant and non-significant effects. The effects appearing beyond the reference line are considered to be significant factors [27]. The Pareto charts rely on the p-value test at > 95% confidence level, displaying the significance of adsorption parameters due to the yield of a wide range of data points. From Fig. 9, it was observed that all of the studied factors (A, B, C and D) which are contact time, pH, sorbent dosage and metal concentration crossed the reference line, therefore they were all significant.

After the significant factors were screened, CCD was used to create a response surface design to evaluate the optimal values of each factor. The yields (optimal values), obtained following performances of the practical experiments were then captured in Table 2.

3.4 Application of Optimal VTrWR to Raw Water Sample

Prior to applying the VTrWR to the raw water samples, profiling for the heavy metal originating from boating pollution was conducted. Fig. 10 shows the concentration of the metals in water samples collected from four areas with major boating and jetty activities.

![Volume of vinegar needed to optimally valorize Tilapia ruweti scale remains to achieve maximal efficiency](image1)

![XRD diffractogram for the VTrWR](image2)
Fig. 7. XRD diffractogram for the non-VTrWR

Fig. 8. FTIR spectra of the fish scale VTrWR and non-VTrWR

Table 2. A summary optimal values for the parameters affecting adsorption efficiency of the VTrWR

<table>
<thead>
<tr>
<th>Ions</th>
<th>pH</th>
<th>Sorbent dosage (mg)</th>
<th>Contact time (min)</th>
<th>Metal concentration (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cd</td>
<td>8.27</td>
<td>80.64</td>
<td>63.89</td>
<td>25.06</td>
</tr>
<tr>
<td>Co</td>
<td>7.68</td>
<td>80.64</td>
<td>76.66</td>
<td>27.43</td>
</tr>
<tr>
<td>Cu</td>
<td>8.07</td>
<td>78.82</td>
<td>72.1</td>
<td>23.85</td>
</tr>
<tr>
<td>Pb</td>
<td>7.97</td>
<td>72.44</td>
<td>88.63</td>
<td>28.44</td>
</tr>
<tr>
<td>Zn</td>
<td>8.75</td>
<td>84.29</td>
<td>69.37</td>
<td>27.49</td>
</tr>
<tr>
<td>Ni</td>
<td>7.19</td>
<td>76.18</td>
<td>52.96</td>
<td>26.06</td>
</tr>
<tr>
<td>All at once</td>
<td>7.19</td>
<td>76.17</td>
<td>72.96</td>
<td>26.06</td>
</tr>
</tbody>
</table>
**Fig. 9.** Pareto charts of standardized effects of the selected ions for the VTrWR

**Fig. 10.** Metal concentration from four sampling points in Okavango Delta
Concentration of metals were higher in areas with boating activities and jetties (Shakawe, Mboma and Xakanaxa) compared to Boro where there are no such activities. Cobalt was not detected while zinc and copper in the water samples were within the acceptable guideline limits for drinking and surface water set out by WHO [28]. However, the concentration of the other selected metals (nickel, lead and cadmium) were above the WHO acceptable limits for drinking water but were within acceptable limits for surface water regulations as shown in the Table 3.

To evaluate the adsorption efficiency of the VTrWR, it was applied to the raw water samples as described in Section 2.10. The percentage ions removals were calculated by employing Equation 1. From the values obtained, a bar graph of percentage removal for the selected ions was constructed. The VTrWR showed high percentage ion removal towards the selected ions; ranging from 60% to 96% from water samples collected from the selected sampling sites as shown in the Fig. 11.

Table 3. A summary of the minimum acceptable limits of the selected ions in drinking and surface waters as set by WHO

<table>
<thead>
<tr>
<th>Metals</th>
<th>Drinking water limits (WHO)</th>
<th>Surface water limits (WHO)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn</td>
<td>3 mg/L</td>
<td>-</td>
</tr>
<tr>
<td>Cd</td>
<td>2 mg/L</td>
<td>-</td>
</tr>
<tr>
<td>Ni</td>
<td>0.02 mg/L</td>
<td>0.05 mg/L</td>
</tr>
<tr>
<td>Pb</td>
<td>0.01 mg/L</td>
<td>0.05 mg/L</td>
</tr>
<tr>
<td>Cd</td>
<td>0.003 mg/L</td>
<td>0.005 mg/L</td>
</tr>
</tbody>
</table>

The efficiency of VTrWR to remove heavy metals from aquatic solution was compared with other adsorbents such as ion imprinted polymers. Table 4 highlights the advantages of employing VTRWR for adsorption of heavy metals compared to use of IIP from our previous work [29].

Fig. 11. Percentage ions removal employing the VTrWR

Table 4. Comparing the use of VTrWR sorbent to IIPS

<table>
<thead>
<tr>
<th>VTrWR</th>
<th>Ion Imprinted Polymers (IIPS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Emphasizes on waste valorization</td>
<td>Produces toxic sludge waste</td>
</tr>
<tr>
<td>Employs green technology</td>
<td>Method uses synthetic polymers and organic solvents</td>
</tr>
<tr>
<td>Its non-selective to pollutants</td>
<td>Tailor made for specific analytes</td>
</tr>
<tr>
<td>It’s cheap and easily accessible</td>
<td>It is expensive to produce the sorbents</td>
</tr>
<tr>
<td>Environmentally friendly</td>
<td>Not environmentally friendly</td>
</tr>
<tr>
<td>It is user friendly</td>
<td>Require technical know how to use</td>
</tr>
<tr>
<td>High adsorption efficiencies (60 to 96%)</td>
<td>High adsorption efficiencies &gt;92%</td>
</tr>
</tbody>
</table>
4. CONCLUSION

The main objectives of this research which were to identify a waste material that were abundant and easily available in our locality, Botswana, valorize the waste materials, thereafter characterize and evaluate the valorized waste materials’ (VTrWR) efficiency in removing the selected toxic ions from surface water of Okavango delta, were successfully achieved as evident from the obtained high average percentage removal of 94.21% for the selected ions by the VTrWR employing optimized conditions. The achieved high % removal by the identified VTrWR was attributed to the functional groups existent on the structures of the waste material which was exposed and made even more accessible to bonding the ions by a simple eco-friendly valorization strategy that involved pulverization and treatment of the waste materials with 12.6% vinegar. Several functional groups including carboxylic, hydroxyl and carbonyls that are known to bind with ions were revealed by Fourier transform infrared (FTIR). Scanning electron microscope (SEM) micrographs showed cylindrical geometry and rough morphology of the VTrWR, which are excellent features in superior sorbents.

In conclusion, the proposed VTrWR with its high % removal efficiency, low cost (since they are collected from waste) and their environmental friendliness (since they are bio-materials thus bio-degradable) presented themselves as potential replacements of the expensive, non-environmentally friendly, commercial adsorbents that are commonly employed for the removal of toxic ions from surface water. Therefore, the use of VTrWR can be adapted by boat owners, riparian communities and relevant stakeholders to purify surface water for environmental conservation as the procedure proved to be simple, un-expensive and user-friendly.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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