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Impact Sound Insulation and Viscoelastic Properties of Resilient Materials made from Recycled Tyre Granules

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The disposal of used tyres is a significant environmental problem in developed countries. The production of acoustic materials from rubber crumbs can therefore represent a valid alternative to incineration or to the disposal of used tyres into landfill.

The goal of the paper is to analyze and optimize the manufacturing process of impact sound insulating materials made of recycled tyre granules mixed with binders. Several prototypes were manufactured in laboratory by means of the developed process and tested in order to evaluate their dynamic stiffness. The influence of grain size, binder concentration and density of the sample was investigated. Thanks to the measured values of the viscoelastic properties, it was possible to model the new materials and to estimate the indexes of impact sound reduction $\Delta L_{W}$. Finally, the prototypes showing the best properties were produced in bigger size and tested in two overlapping reverberating rooms according to the standard ISO 140-8. Results confirm that the materials manufactured through the developed process have satisfactory acoustic properties, comparable to the ones of commercially available materials.

1. INTRODUCTION

Energy consumption in the building sector can reach up to 40% of the total energy demand of an industrialized country. For this reason, green building strategies can be extremely effective as far as fossil fuel savings and greenhouse gas reduction. Sustainable materials made from natural or recycled materials can play an important role, since less energy is generally required for their production than that needed for producing conventional materials.

In the last years a great attention has been focused on green materials, especially in the building sector. Many research centres have developed new sustainable materials, in many cases with interesting thermal and acoustical properties.\(^1\) In addition, the public sector has started to consider these materials. In Italy, for instance, many municipalities have introduced specific recommendations into building regulations to increase the use of ecological materials in new constructions, allowing a reduction of construction taxes.

In particular, natural fibres are increasingly considered as alternatives to synthetic ones, in order to combine high acoustic and thermal performance with a low impact on the environment and human health. Recycled materials, such as recycled plastic fibres and recycled rubber mats, can even be regarded as a sustainable alternative, as they contribute to lower waste production and use of raw materials.

Within this context, recycled tyre granules have been recently proposed for manufacturing acoustic insulating and absorbing materials, to be used for noise control in buildings and road barriers. Materials made of rubber crumbs usually have high porosity, and they consequently show good sound absorbing properties over a wide frequency range. Furthermore, the use of these materials in insulating underlays shows satisfactory performance for acoustic impact insulation.

The development of novel acoustic materials made from end-of-life tyres can possibly be a solution for the disposal of these materials. Used tyres represent in fact a huge amount of material to be disposed. Estimates are that more than 250 million post-consumer tyres are accumulated each year in the fifteen Member States of the European Union, and comparable amounts are amassed in Eastern Europe, North America, Latin America, Asia, and the Middle-East, totalling more than 1,000 million additional tyres per year.\(^2\) The largest reuse market is tyre-derived fuel, followed by civil engineering applications such as highway embankments; however, the production of rubber crumbs is growing, and the subsequent applications are gaining importance.\(^3\)

The goal of this paper is to develop an innovative process using recycled tyre granules to produce materials for acoustic applications, in particular resilient resilient materials, made from tyre granules and presents the chemical characterization of the materials, the description of the manufacturing process, and the results of the experimental campaign and modelling process carried out at the Acoustic Laboratory of the University of Perugia.
2. LITERATURE REVIEW

Several authors studied the acoustic properties of materials made from rubber crumbs. Most of the research found in literature focuses mainly on the acoustic absorption properties of these materials. Pfeitzschener and Rodriguez demonstrated among the firsts that recycled rubber products show excellent performance of broadband acoustic absorption. Horoshenkov and Swift provided a comprehensive description of the sound absorption properties of both loose and consolidated rubber crumbs. Sobral et al. correlated the sound absorption properties of bound rubber crumbs to the mechanical ones. More recently, Hong et al. studied improved acoustic performance by combining double-layer structures of rubber particles with porous materials and perforated panels, obtaining satisfactory values of the absorption coefficient.

Although many studies found in literature deal with the prediction and measurement of the damping properties of rubber-based materials, the ones concerning rubber crumbs from recycled tyres are much more limited, so there is a need to fill a gap in this research area. As a matter of fact, there is a certain interest of the tyre manufacturers toward these materials. An important Italian company in the field of tyres has recently developed a pre-mixed material from end-of-life tyre crumbs with a water-based binder that can be laid as a normal screed, showing good impact sound insulation properties.

A research study dealing with impact sound reduction of underlays made of recycled materials has been conducted by Rusforth et al. concerning carpet wastes. The material is composed by a fibrous and a granular part and has completely different properties compared to rubber crumbs, but the metrological approach and results are interesting for the current subject.

Previous works by the authors evaluated the dynamic stiffness of rubber crumb underlays to be used in buildings for impact sound reduction, and this research obtained encouraging results.

3. MATERIALS PRODUCTION AND CHARACTERIZATION

Thanks to the instrumentation available at the European Center of Nanostructured Polymers of the University of Perugia, several analyses were performed to investigate the main physical and chemical characteristics of the rubber grains derived from scrap tyres, and in particular, the analyses consisted of grain size analysis, thermo-mechanical and thermo-gravimetric characterization, and differential scanning calorimetry.

The composition of the rubber was studied by means of a SEM-FEG (Field Emission Gun Scanning Electron Microscope) equipped with an EDX (Energy Dispersive X-ray spectroscopy) analysis system. The results in terms of percentage in weight of the chemical elements are 92.0 % of Carbon, 4.9 % of Oxygen, 1.6 % of Sulphur and the 1.5 % of Zinc. The content of hydrogen cannot be measured with this technique.

Tyre crumbs were provided by a company owning grinding machines and sieves.

Different grain sizes were available (from a thin powder to bigger pieces), but only two of them were considered suitable for the required applications: a small size (average diameter of the granules = 1–2 mm) and a medium size (3–5 mm). Larger-sized grains (6–10 mm) were not used in order to achieve a good homogeneity and thickness. (See also, section 5.)

As far as the selection of the binder, previous studies by the authors showed that the most suitable binder for this application is a prepolymerized polyurethane. A polyurethane binder suggested by a partner chemical company was used for the present activity because of its particular attitude to tie granular materials, in particular rubber crumbs.

An analysis with a rheometer was performed in order to identify the optimal condition for mixing the rubber crumbs and the binder. The lowest value of viscosity of the binder, which implies the highest fluidity, is reached when the temperature is 55 °C. Of course, a very fluid binder can optimally cover the granules and bind them together.

The manufacturing process can be summarized as follows.

The first steps are weighing the rubber crumbs by means of a precision balance and heating the binder to 55 °C, the condition for lowest viscosity. Subsequently, the binder is weighted and added to the rubber by means of a planetary mixer.

When the rubber crumbs are uniformly covered by the binder, the mixture is put in a 26 x 26 cm square die made of steel. The die is then placed in a hot press for 15 minutes at 130 °C and 10 bar. Finally, the sample is removed from the die and can be tested after at least 48 hours. Figure 1 reports some pictures taken during different stages of the manufacturing process.

4. PRELIMINARY RESULTS

In a previous experimental campaign, six samples were prepared and tested at the Acoustics Laboratory of the University of Perugia in order to evaluate the influence of some characteristics of the materials (e.g., grain size, consolidation ratio, binder concentration) on the elastic and acoustic properties in terms of dynamic stiffness. The samples were manufactured by means of a simple cold process using the tools available at the Acoustics Lab. The rubber crumbs and the binder were mixed with a commercial cooking mixer and left under a weight for consolidating. Table 1 reports the main characteristics and the measured values of the dynamic stiffness of the samples.

Consolidation ratio is defined as the ratio between the variation of thickness after the consolidation process and the starting unconsolidated thickness. The binder concentration is related to the percentage in weight of the binder contained in the mix.

The samples made with larger grains show the best acoustic properties because of the inner elasticity of the rubber (in Table 1 see G1 vs. GM); moreover, they do not need to be consolidated. On the other hand, layers made only with the large grain size (size 6–10 mm) have a large number of hollows. Furthermore, the thickness of the layer depends on the size of the grains. Therefore, it is impossible to reach thickness
values lower than the size of the granules. For such a reason, larger grains have been excluded in the following analysis.

The comparison of the results obtained for the samples M1 and M2 shows that more consolidated samples have higher dynamic stiffness. However, a certain consolidation is needed to guarantee satisfactory mechanical properties.

The results of the samples G1, G2 and G3 seem to suggest that an increase of the binder concentration causes an increase of the dynamic stiffness of the sample, but the characteristics and the type of the binder greatly influence this effect.

5. EXPERIMENTAL CAMPAIGN

Two stages of experimental tests were executed. The first session aimed at identifying the optimal characteristics of the materials in terms of grain size and mix, grain density, and binder concentration. In this stage, dynamic stiffness measurements were carried out according to EN 29052-1 standard on several small-size prototypes (20 x 20 cm).\textsuperscript{15}

Dynamic stiffness is a fundamental parameter for resilient insulating layers since it directly influences the efficiency of the material in impact noise damping.

The sample being tested is placed under a steel load plate acting as the load mass (weight = 8 kg). A layer of plaster is applied on the surface of the sample in order to cover all the irregularities; after the time of curing (about 3 days), the test can be executed. Tests are executed by placing the plate and the sample on the floor in a reverberating room in order to get rid of external disturbance (vibrations, steps, etc.).

The dynamic stiffness is evaluated by measuring the resonance frequency of the mass-spring-mass system, with the load plate, resilient layer, and floor of the reverberating room as these respective components. Force is applied by means of a shaker fed by a sweep signal, while the frequency resonance is evaluated by measuring the acceleration versus the variation of the excitation frequency. The accelerometer and the load cell are produced by Dytran Instrument Inc., the shaker by Wilcoxon Research, and the charge amplifier and the software by Microbel. The measurement uncertainty of the system is always lower than 1%.

As noted by Baron et al., it is important to excite the plate and measure the response in the vicinity of its center in order to avoid the generation of flexural modes.\textsuperscript{16}

After choosing the best performing sample, a second session of measurements was performed according to ISO 140-8 standard on a larger-sized sample (100 x 100 cm), by means of two overlapping reverberation rooms in order to evaluate the impact sound reduction $\Delta L$.\textsuperscript{17}

5.1. First Stage: Dynamic Stiffness Measurements

The production of the prototypes was designed taking into account the preliminary results and the evidences coming from other studies in order to optimize the values for grain mix, grain density, and binder concentration for applications as impact insulators.

To this end, seventeen typologies of prototypes were manufactured; three samples of each kind were produced (for a total of fifty-one samples) and the average results are reported in the following sections. The thickness of the samples was 8 mm, a common value for commercial resilient layers.

The lateral flow resistivity of the samples tested was not measured, but other authors have found values always lower than 10 kPa * s/m² for such layers.\textsuperscript{5,6} According to EN 29051-1, in this case the apparent dynamic stiffness $\sigma_1$ coincides with the dynamic stiffness $s$.

The impact sound reduction index $\Delta L_W$ was estimated from the values of dynamic stiffness according to EN 12354-2 stan-
Figure 2. Effect of the grains' mix on the resonance frequency $f_r$ and dynamic stiffness $s$ of the material.

Figure 3. Effect of the density on the resonance frequency $f_r$ and dynamic stiffness $s$ of the material.

In order to have a simple and immediate term of comparison.

5.1.1. Optimization of the Grains' Mix

As previously said in section 5.1.1, two grain sizes were considered suitable for the required applications: a "small" size (average diameter of the granules = 1–2 mm, indicated in the following with the initial S) and a "medium" size (average diameter of the granules = 3–5 mm, indicated with the initial M). The sizes are given by the sieves used for the separation of the crumbs.

Five grain mixes were selected to be tested, keeping constant the binder concentration of 13%: (1) 100 % M, (2) 80 % M + 20 % S, (3) 50 % M + 50 % S, (4) 20 % M + 80 % S, and (5) 100 % S. The results in terms of resonance frequency $f_r$ and dynamic stiffness $s$ are reported in Fig. 2. The best performance was achieved by the sample made of 80 % small and 20 % medium grains; consequently, this mix was considered optimal and chosen for the continuation of the experimentation.

5.1.2. Optimization of the Density

In this stage, six prototypes were realized with the same binder concentration (13 %) and mix of the grain sizes (80 % M + 20 % S), changing the quantity of the compound rubber-binder put in the mould (from 292 g to 405 g), keeping constant the size and final thickness of the layer. In this case, it is therefore not possible to refer to consolidation ratio. Higher/lower density was discarded as the corresponding samples were too hard/breakable. The results are reported in Fig. 3.

As expected, denser (i.e., more consolidated) materials showed the worst performance because of the excessive hardening of the solid matrix, while the samples with intermediate density (between 579 and 697 kg/m$^3$) had the best elastic characteristics (dynamic stiffness between 61 and 68 MN/m$^3$).

5.1.3. Optimization of the Binder Concentration

The binder concentration is defined as the ratio between the mass of the binder and the total mass of the compound rubber-binder multiplied by 100 and expressed as a percentage.

For the samples tested, it was observed that the optimal density was between 579 and 697 kg/m$^3$. For this reason, two sessions of measurements were performed, with two series of samples having density respectively of 579 and 697 kg/m$^3$.

The mix of the grain sizes was always 80 % M + 20 % S.

For the first session (density = 697 kg/m$^3$), four prototypes were produced with the following binder concentrations: 4.8 %, 7.0 %, 10.3 % and 13.0 %. The results are reported in Fig. 4.

The dynamic stiffness decreases almost linearly with increasing values of the binder concentrations, confirming the good elastic properties of the chosen polyurethane binder.

Five prototypes were tested in the second session. The density of the samples was 579 kg/m$^3$, while the binder concentration was 9.1 %, 11.1 %, 13.0 %, 14.9 %, and 16.7 %. The results are reported in Fig. 5. In this case, the best performance is achieved by samples with binder concentrations equal to 13.0 % and 16.7 %.
Table 2. Physical properties and acoustic performance of the tested samples.

<table>
<thead>
<tr>
<th>ID</th>
<th>Grains’ mix</th>
<th>Density [kg/m³]</th>
<th>Binder Conc. [%]</th>
<th>Avg. resonance frequency [Hz]</th>
<th>Quality factor Q</th>
<th>Damping ratio [δ [%]]</th>
<th>Dynamic stiffness [MN/m³]</th>
<th>ΔLW [dB]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100 % S</td>
<td>636</td>
<td>13</td>
<td>104.5</td>
<td>8.3</td>
<td>12.0</td>
<td>86</td>
<td>23.5</td>
</tr>
<tr>
<td>2</td>
<td>20 % M 80 % S</td>
<td>717</td>
<td>13</td>
<td>109.4</td>
<td>8.9</td>
<td>11.2</td>
<td>94</td>
<td>22.9</td>
</tr>
<tr>
<td>3</td>
<td>50 % M 50 % S</td>
<td>614</td>
<td>13</td>
<td>94.4</td>
<td>10.9</td>
<td>9.2</td>
<td>71</td>
<td>24.8</td>
</tr>
<tr>
<td>4</td>
<td>80 % M 20 % S</td>
<td>697</td>
<td>13</td>
<td>92.5</td>
<td>11.4</td>
<td>8.8</td>
<td>68</td>
<td>25.1</td>
</tr>
<tr>
<td>5</td>
<td>100 % M</td>
<td>645</td>
<td>13</td>
<td>95.7</td>
<td>10.1</td>
<td>9.9</td>
<td>73</td>
<td>24.7</td>
</tr>
<tr>
<td>6</td>
<td>80 % M 20 % S</td>
<td>540</td>
<td>13</td>
<td>100.7</td>
<td>8.6</td>
<td>11.6</td>
<td>80</td>
<td>24.0</td>
</tr>
<tr>
<td>7</td>
<td>80 % M 20 % S</td>
<td>579</td>
<td>13</td>
<td>88</td>
<td>7.9</td>
<td>12.6</td>
<td>61</td>
<td>25.7</td>
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<tr>
<td>8</td>
<td>80 % M 20 % S</td>
<td>616</td>
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<td>90.7</td>
<td>8.3</td>
<td>12.0</td>
<td>65</td>
<td>25.4</td>
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<tr>
<td>9</td>
<td>80 % M 20 % S</td>
<td>723</td>
<td>13</td>
<td>135.7</td>
<td>12.0</td>
<td>8.3</td>
<td>145</td>
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<tr>
<td>10</td>
<td>80 % M 20 % S</td>
<td>749</td>
<td>13</td>
<td>131.5</td>
<td>10.4</td>
<td>9.6</td>
<td>137</td>
<td>20.5</td>
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<tr>
<td>11</td>
<td>80 % M 20 % S</td>
<td>697</td>
<td>4.8</td>
<td>107.7</td>
<td>6.4</td>
<td>15.6</td>
<td>92</td>
<td>23.1</td>
</tr>
<tr>
<td>12</td>
<td>80 % M 20 % S</td>
<td>697</td>
<td>7</td>
<td>104.8</td>
<td>9.2</td>
<td>10.9</td>
<td>87</td>
<td>23.5</td>
</tr>
<tr>
<td>13</td>
<td>80 % M 20 % S</td>
<td>697</td>
<td>10.3</td>
<td>97</td>
<td>8.7</td>
<td>11.5</td>
<td>75</td>
<td>24.5</td>
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<tr>
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<td>80 % M 20 % S</td>
<td>579</td>
<td>9.1</td>
<td>99.6</td>
<td>9.0</td>
<td>11.1</td>
<td>78</td>
<td>24.1</td>
</tr>
<tr>
<td>15</td>
<td>80 % M 20 % S</td>
<td>579</td>
<td>11.1</td>
<td>94.1</td>
<td>10.2</td>
<td>9.8</td>
<td>70</td>
<td>24.8</td>
</tr>
<tr>
<td>16</td>
<td>80 % M 20 % S</td>
<td>579</td>
<td>14.9</td>
<td>90.5</td>
<td>8.3</td>
<td>12.1</td>
<td>66</td>
<td>25.5</td>
</tr>
<tr>
<td>17</td>
<td>80 % M 20 % S</td>
<td>579</td>
<td>16.7</td>
<td>87.2</td>
<td>9.2</td>
<td>10.9</td>
<td>61</td>
<td>25.9</td>
</tr>
</tbody>
</table>

5.2. Evaluation of the Reduction in Normalized Impact Sound Pressure

As it is well known, the index of impact sound reduction ΔLW can be estimated knowing the dynamic stiffness by means of the equation given by Brocco:19

\[ ΔL_W = 18 + 15 \log \frac{m'}{\delta'} \]  \hspace{1cm} (1)

where \( m' \) is the mass per unit area of the floating floor in kg/m². Another important parameter that has to be considered is the damping ratio \( \delta \), defined as the inverse of the quality factor \( Q \):

\[ \delta = \frac{1}{Q} \]  \hspace{1cm} (2)

and

\[ Q = \frac{f_2}{f_2 - f_1} \]  \hspace{1cm} (3)

where \( f_s \) is the measured resonance frequency of the rubber layer and \( f_1 \) and \( f_2 \) are the values of the frequency on the resonant curve at -3 dB of the resonance frequency (see Fig. 6).

In Table 2, the following parameters are reported for all the tested samples (with average values, since each test was executed on three specimens): manufacturing parameters, resonance frequency, quality factor, damping factor, dynamic stiffness, and the estimated index of impact sound reduction ΔLW.

As expected the manufactured layers have low values of the damping ratio (\(< 15 \%\)), due to the intrinsic high value of the elasticity coefficient of the rubber.

In Table 2 the best performing sample (number 7) is highlighted; its characteristics are: mix of the grains’ size: 80 % medium size and 20 % small size; binder concentration: 13.0 %; density: 579 kg/m³; weight: 313 g.

![Resonant curve with the indication of \( f_s, f_1, f_2 \) values.]

This sample was chosen as the optimal one because of its values of dynamic stiffness (one of the lowest) and of damping ratio (one of the highest). Also, sample 17 has a similar performance, but with a higher binder concentration, which means a higher cost and environmental impact.

As stated by Pavoni Belli et al., the accuracy of the estimation of ΔLW decreases with the decrease of the damping ratio, in this case, decreasing quite low.20 For this reason a laboratory measurement of the reduction of impact sound pressure has been carried out to validate the measurement of dynamic stiffness.

5.3. Second Stage: Impact Sound Reduction Measurements

The second stage aimed at evaluating the acoustic performance of the optimized underlay by measuring the impact sound reduction ΔL in two overlapping reverberation rooms according to ISO 140-8. It was impossible to produce a larger sample (100 x 100 cm vs. 20 x 20 cm needed for dynamic stiffness measurements) with the manufacturing process described in Section 5.1.1. For this reason, sixteen square 25 cm lay-
ers were laid on the floor of the upper reverberation room and joined with tape. A 6 cm thick homogeneous concrete slab was put on the sample (see Fig. 7).

The tapping machine was placed in three positions on the sample and three positions on the bare floor around the sample; the measurement was then repeated moving the sample in a different position of the floor of the upper reverberation room. Six microphone positions were used in the receiving room for a total of seventy-two acquisitions. Also, background noise was measured, but no correction was needed.

Figure 8 reports the third-octave bands trend of the impact sound reduction $\Delta L$. The index of impact sound reduction $\Delta L_W$ calculated according to ISO 717-2 is 26 dB, in perfect agreement with the value estimated from the dynamic stiffness (25.7 dB).\textsuperscript{21}

The obtained value compares favourably with some commercial products, especially with other rubber crumb underlays, as shown in Table 3.

### 6. CONCLUSIONS

Used tyres represent a serious environmental problem since it is no longer possible to dispose them into landfills in the EU; also, the other traditional use of end-of-life tyres – their incineration in concrete production plants – causes a high environmental impact. The use of rubber crumb made from end-of-life tyres to produce damping materials for buildings applications can therefore represent a sustainable solution.

In recent years, many research laboratories have studied, both from a theoretical and an experimental point of view, new acoustic materials made of recycled fibres and grains. This paper presents the results of an experimental campaign carried out in the Acoustics Lab of the Department of Industrial

<table>
<thead>
<tr>
<th>Manufactured sample</th>
<th>$\Delta L_W$ (dB)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regupol\textsuperscript{1}</td>
<td>28</td>
<td>8</td>
</tr>
<tr>
<td>Pavigran Estra\textsuperscript{*}</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>Pavigran RC\textsuperscript{*}</td>
<td>19</td>
<td>4</td>
</tr>
<tr>
<td>Recycled polyurethane with polyethylene film</td>
<td>17</td>
<td>6</td>
</tr>
<tr>
<td>Closed cells expanded polyethylene</td>
<td>27</td>
<td>10</td>
</tr>
<tr>
<td>Heat-bonded polyester fibers</td>
<td>27</td>
<td>10</td>
</tr>
<tr>
<td>Closed cells polyolefin</td>
<td>30</td>
<td>7</td>
</tr>
</tbody>
</table>

\textsuperscript{*} Commercial name

![Figure 7. From left to right: the tested acoustic underlay, the concrete slab on the sample, and the tapping machine.](image)

![Figure 8. Impact sound reduction of the sample vs. frequency.](image)

Engineering of the University of Perugia, and focuses on the impact sound insulation properties of various samples made of tyre granules, bounded with an appropriate binder.

A wide campaign of dynamic stiffness measurements was carried out. The influence on the impact sound insulation properties (e.g., granule size, binder concentration, sample thickness, sample compaction ratio) was investigated, and a modelling and optimization process was carried out. The results on 8 mm thick samples made of medium and large size tyre granules with a polyurethane binder show good performance ($s = 61 \text{ MN/m}^3$, with a measured $\Delta L_W = 26 \text{ dB}$), similar to the performance of commercially available rubber made materials of the same thickness.

The research will therefore continue with the production and testing of larger samples, together with the measurement of compressibility and compressive creep, in order to evaluate long term acoustical performance.\textsuperscript{22}
7. ACKNOWLEDGEMENTS

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