# Supplementary Material

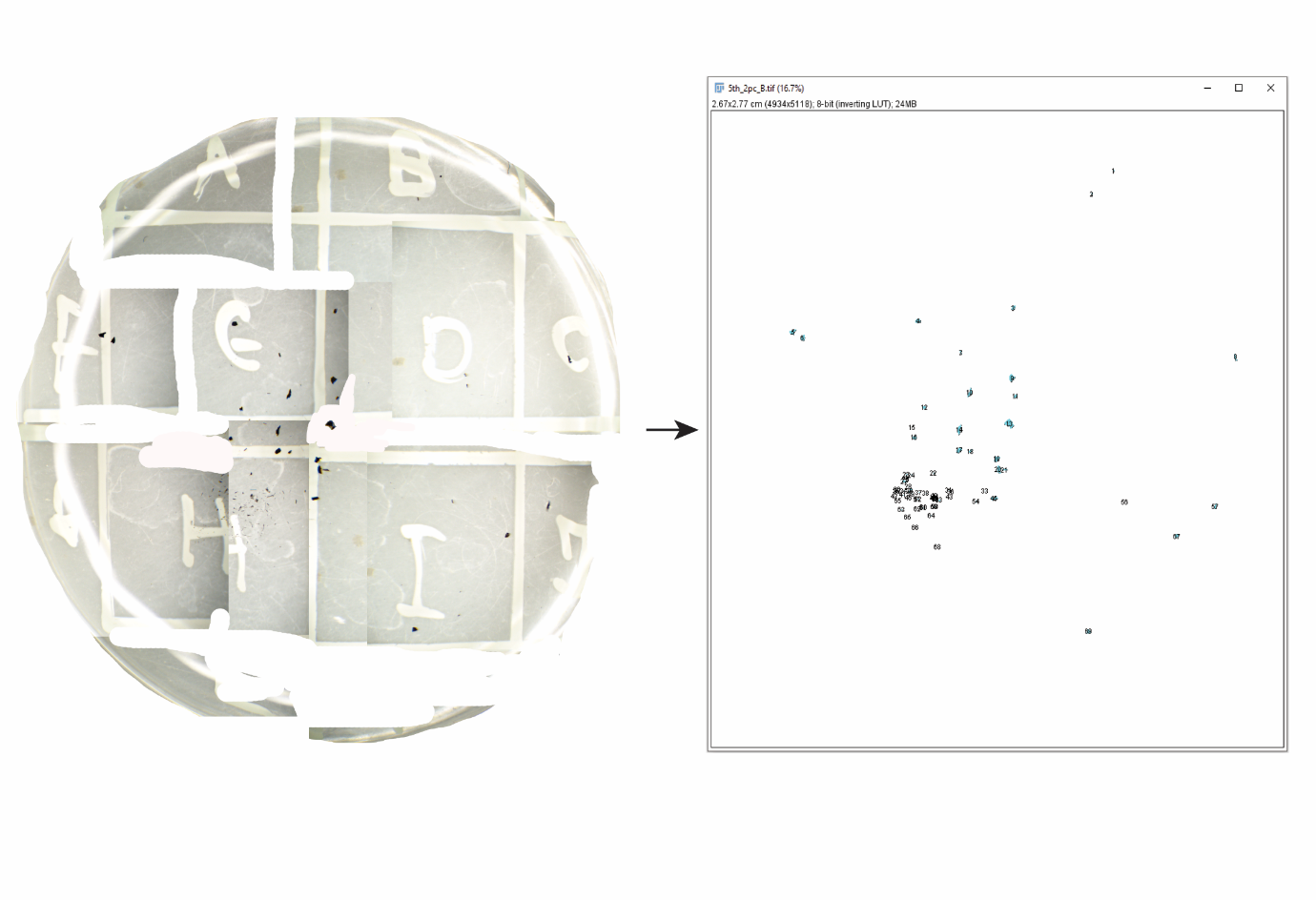


Figure S1. Reconstructed petri dish of NaClO 12.5% at the final time step (T4) and automated identification of charcoal fragments in ImageJ.

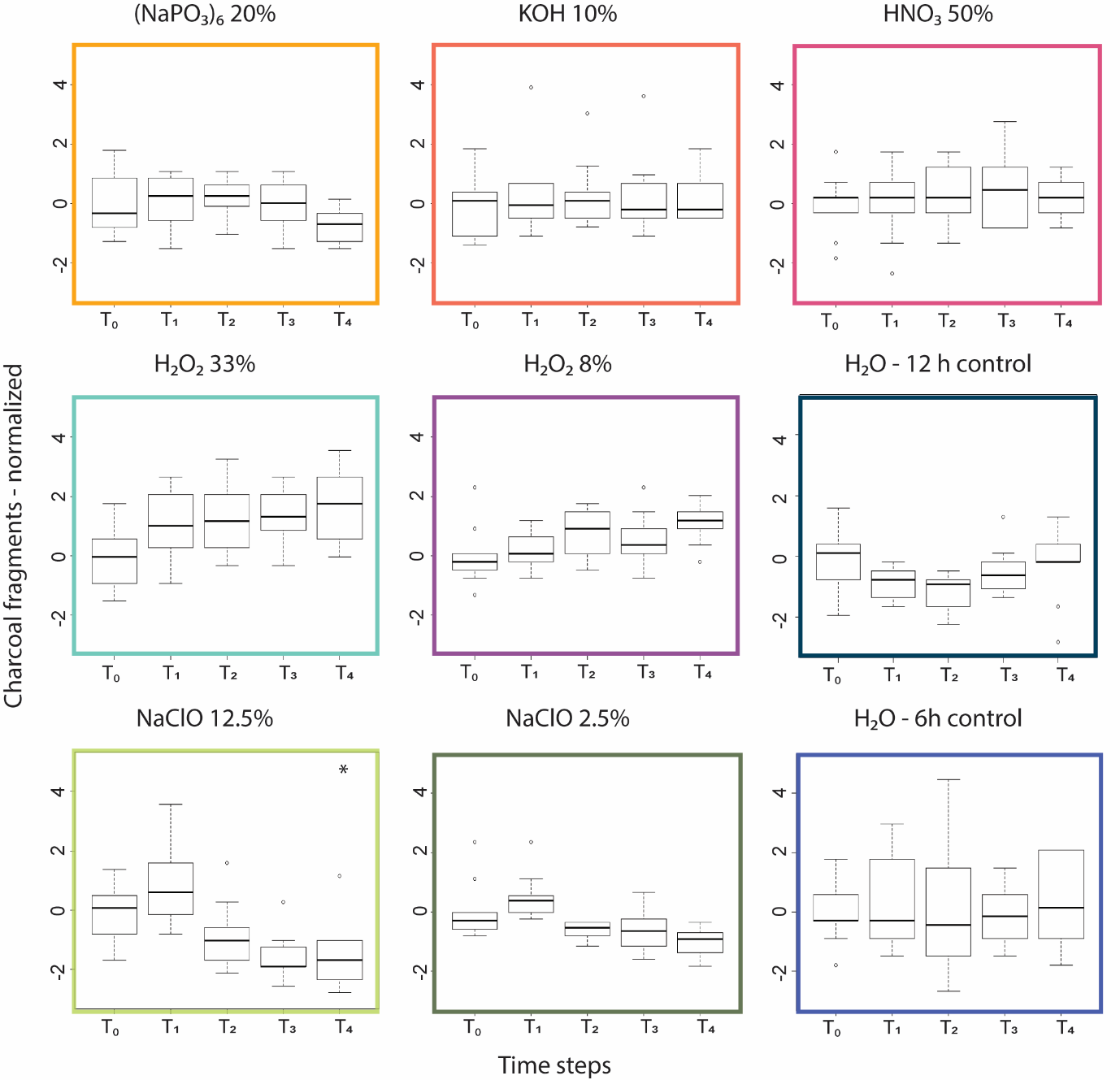


Figure S2. Charcoal number of fragments change over 12h and 6h time steps when subjected to widely used chemicals in charcoal analysis. Samples constitute a known mixture of modern charcoal types produced in the lab. For every chemical treatment and two H2O control sets n = 10. Mixed ANOVAs were not possible for this data set due to violations on the assumption of sphericity. Color boxes are the same used as in Figures 2 and 3 to indicate the same treatments.

Table S1: Chemicals, concentrations, and exposure times used for the extraction of charcoal for fire studies around the world. More than one chemical usually indicates a second bleaching step. For full names of the chemicals see text. n/a = not available information in the referenced publication. Note this is not an exhaustive list. Studies were chosen on the basis that they were from all around the world and using different chemicals or combinations thereof.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Reference | Chemical | Conc. | Time | Sediment type | Country | Notes |
| Blarquez et al., 2018 | NaClO | 8% | 24 h | Peat | Canada |  |
| Hawthorne and Mitchell, 2016 | (NaPO3)6  H2O2 | 15%  diluted | 24 h  n/a | Lacustrine | Ireland |  |
| Crawford and Belcher, 2016 | NaClO | 8% | 20 h | Peat | UK |  |
| Fletcher et al., 2018 | NaClO | n/a | n/a | Lacustrine | Tasmania |  |
| Mariani and Fletcher, 2017 | NaClO | 5% | <2 weeks | Multiple | Australia |  |
| Walsh et al., 2017 | (NaPO3)6  NaClO | 5%  weak | >24 h  1h | Lacustrine | USA |  |
| Finsinger et al., 2017 | NaOH  NaClO | 10%  2% | n/a  n/a | Lacustrine | Romania |  |
| Anderson and Wahl, 2016 | (NaPO3)6  NaClO | 3%  6% | >24 h  n/a | Lacustrine | Guatemala |  |
| Pérez -Obiol et al., 2016 | KOH  NaClO | n/a  15% | 1.5 h  1.5 h | Peat | Spain | samples heated to 70°C |
| Chipman et al., 2015 | NaO3P  NaClO | 10%  n/a | 20h  20h | Lacustrine | Alaska, USA | freeze-dried overnight / chemicals possibly mixed |
| Robin et al., 2013 | NaClO | 13% | 24 h | Peat | Germany |  |
| Genries et al., 2012 | NaP2O4  NaClO | 3%  10% | n/a  n/a | Lacustrine | Canada |  |
| Olsson et al., 2010 | NaClO | 5% | 24 h | Lacustrine | Sweden |  |
| Gardner and Whitlock, 2001 | (NaPO3)6  NaClO | 1%  5% | 24 h  5min | Lacustrine | Northwest USA | heated at low temperature to remove excessive invertebrate faeces |
| Glais et al., 2017 | NaOH  H2O2 | 10%  6% | 24 h  24 h | Peat | Greece |  |
| Finsinger et al., 2017 | H2O2 | 15% | overnight | Peat | Serbia |  |
| Spencer et al., 2017 | H2O2 | 3% | n/a | Lacustrine | USA |  |
| Pillai et al., 2017 | NaO3P  H2O2 | 5%  8% | n/a  12h | wetland cores | India |  |
| Colombaroli et al., 2018 | NaO3P  H2O2 | 5%  8% | n/a  12h | Lacustrine | Kenya |  |
| Rius et al., 2011 | NaOH  H2O2 | 10%  6% | 24 h  24 h | Multiple | France |  |
| Mustaphi and Pisaric, 2014 | NaO3P | n/a | 24 h | Lacustrine | Canada |  |
| Miyabuchi et al., 2012 | (NaPO3)6  KOH  H2O2 | 5%  10%  1% | 24 h  24 h  n/a | Tephra | Japan |  |
| Olsson et al., 2010 | NaOH | 10% | 24 h | Lacustrine | Sweden |  |
| Thevenon et al., 2003 | HCL  HNO3  H2O2 | 3M  conc.  33% | n/a  n/a  n/a | Lacustrine | Tanzania |  |

Table S2: Mean and standard deviation of normalized replicates within each time step for the 12 h batch. Results of repeated measures ANOVA for the 12h batch—significance of the effect of treatment noted.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Overall | **H2O2 33%** | *2.05±1.19* |  |  |  |  |
|  | **H2O2 8%** | 0.99±0.57 | 0.99±0.57 |  |  |  |
|  | **HNO3 50%** | 1.38±0.81 | 1.38±0.81 | 1.38±0.81 |  |  |
|  | **KOH 10%** | 1.65±1.08 | 1.65±1.08 | 1.65±1.08 | 1.65±1.08 |  |
|  | **(NaPO3)6 20%** | *1.95±1.11* | 1.95±1.11 | 1.95±1.11 | 1.95±1.11 | 1.95±1.11 |
| T0 | **H2O2 33%** | 0±1 |  |  |  |  |
|  | **H2O2 8%** | 0±1 | 0±1 |  |  |  |
|  | **HNO3 50%** | 0±1 | 0±1 | 0±1 |  |  |
|  | **KOH 10%** | 0±1 | 0±1 | 0±1 | 0±1 |  |
|  | **(NaPO3)6 20%** | 0±1 | 0±1 | 0±1 | 0±1 | 0±1 |
| T1 | **H2O2 33%** | 2.01±1.06 |  |  |  |  |
|  | **H2O2 8%** | 1.05±0.71 | 1.05±0.71 |  |  |  |
|  | **HNO3 50%** | 1.42±0.82 | 1.42±0.82 | 1.42±0.82 |  |  |
|  | **KOH 10%** | 1.17±1.22 | 1.17±1.22 | 1.17±1.22 | 1.17±1.22 |  |
|  | **(NaPO3)6 20%** | 2.14±1.14 | 2.14±1.14 | 2.14±1.14 | 2.14±1.14 | 2.14±1.14 |
| T2 | **H2O2 33%** | ***2.79±0.78*** |  |  |  |  |
|  | **H2O2 8%** | 1.43±0.58 | 1.43±0.58 |  |  |  |
|  | **HNO3 50%** | 1.60±0.80 | 1.60±0.80 | 1.60±0.80 |  |  |
|  | **KOH 10%** | 2.00±1.20 | 2.00±1.20 | 2.00±1.20 | 2.00±1.20 |  |
|  | **(NaPO3)6 20%** | *2.68±0.92* | 2.68±0.92 | 2.68±0.92 | 2.68±0.92 | 2.68±0.92 |
| T3 | **H2O2 33%** | ***2.62±0.69*** |  |  |  |  |
|  | **H2O2 8%** | 1.26±0.63 | 1.26±0.63 |  |  |  |
|  | **HNO3 50%** | 1.85±0.86 | 1.85±0.86 | 1.85±0.86 |  |  |
|  | **KOH 10%** | ***2.39±1.14*** | 2.39±1.14 | 2.39±1.14 | 2.39±1.14 |  |
|  | **(NaPO3)6 20%** | *2.56±0.98* | 2.56±0.98 | 2.56±0.98 | 2.56±0.98 | 2.56±0.98 |
| T4 | **H2O2 33%** | ***2.81±0.64*** |  |  |  |  |
|  | **H2O2 8%** | 1.21±0.42 | **1.21±0.42** |  |  |  |
|  | **HNO3 50%** | 2.04±0.82 | 2.04±0.82 | *2.04±0.82* |  |  |
|  | **KOH 10%** | *2.67±1.29* | 2.67±1.29 | 2.67±1.29 | 2.67±1.29 |  |
|  | **(NaPO3)6 20%** | 2.39±1.29 | 2.39±1.29 | 2.39±1.29 | 2.39±1.29 | 2.39±1.29 |
|  |  | **H2O 12h** | **H2O2 33%** | **H2O2 8%** | **HNO3 50%** | **KOH 10%** |

p>0.5; *p<0.05*; ***p<0.01***; **p<0.001**

Table S3: Mean and standard deviation of normalized replicates within each time step for the 6 h batch. Results of repeated measures ANOVA for the 6h batch—significance of the effect of treatment noted.

|  |  |  |  |
| --- | --- | --- | --- |
| Overall | **NaClO 2.5%** | **0.06±0.62** |  |
|  | **H2O** | **0.24±0.26** | 0.24±0.26 |
| T0 | **NaClO 2.5%** | 0±1 |  |
|  | **H2O** | 0±1 | 0±1 |
| T1 | **NaClO 2.5%** | **0.920±0.42** |  |
|  | **H2O** | **0.500±0.54** | 0.500±0.54 |
| T2 | **NaClO 2.5%** | **0.29±0.66** |  |
|  | **H2O** | ***−0.66±1.13*** | −0.66±1.13 |
| T3 | **NaClO 2.5%** | **−0.12*±0.62*** |  |
|  | **H2O** | **0.47*±1.36*** | 0.47*±1.36* |
| T4 | **NaClO 2.5%** | **−0.79±0.47** |  |
|  | **H2O** | **0.30±0.68** | ***0.30±0.68*** |
|  |  | **NaClO 12.5%** | **NaClO 2.5%** |

p>0.5; *p<0.05*; ***p<0.01***; **p<0.001**

Table S4: Mean and standard deviation of normalized replicates within each chemical for the 12 h batch. Results of repeated measures ANOVA for the 12 h batch—significance of the effect of time noted.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Overall | **T1** | **1.44±0.22** |  |  |  |
|  | **T2** | **1.92±0.21** | **1.92±0.21** |  |  |
|  | **T3** | **1.92±0.19** | **1.92±0.19** | 1.92±0.19 |  |
|  | **T4** | **1.20±0.35** | **1.20±0.35** | 1.20±0.35 | 1.20±0.35 |
| H2O 12h | **T1** | *0.85±0.73* |  |  |  |
|  | **T2** | ***1.05±0.91*** | 1.05±0.91 |  |  |
|  | **T3** | 0.84±0.82 | 0.84±0.82 | 0.84±0.82 |  |
|  | **T4** | *0.88±0.77* | 0.88±0.77 | 0.88±0.77 | 0.88±0.77 |
| H2O2 33% | **T1** | **2.01±1.06** |  |  |  |
|  | **T2** | **2.79±0.78** | *2.79±0.78* |  |  |
|  | **T3** | **2.62±0.69** | 2.62±0.69 | 2.62±0.69 |  |
|  | **T4** | **2.81±0.64** | 2.81±0.64 | 2.81±0.64 | 2.81±0.64 |
| H2O2 8% | **T1** | *1.05±0.71* |  |  |  |
|  | **T2** | **1.43±0.57** | 1.43±0.57 |  |  |
|  | **T3** | **1.26±0.63** | 1.26±0.63 | 1.26±0.63 |  |
|  | **T4** | *1.21±0.42* | 1.21±0.42 | 1.21±0.42 | 1.21±0.42 |
| HNO3 50% | **T1** | **1.42±0.82** |  |  |  |
|  | **T2** | **1.60±0.80** | 1.60±0.80 |  |  |
|  | **T3** | **1.85±0.86** | *1.85±0.86* | 1.85±0.86 |  |
|  | **T4** | **2.04±0.82** | ***2.04±0.82*** | *2.04±0.82* | *2.04±0.82* |
| KOH 10% | **T1** | 1.17*±1.22* |  |  |  |
|  | **T2** | ***2.00±1.20*** | 2.00±1.20 |  |  |
|  | **T3** | **2.39±1.14** | ***2.39±1.14*** | 2.39±1.14 |  |
|  | **T4** | **2.67±1.29** | **2.67±1.29** | *2.67±1.29* | 2.67±1.29 |
| (NaPO3)620% | **T1** | ***2.14±1.14*** |  |  |  |
|  | **T2** | **2.68±0.92** | 2.68±0.92 |  |  |
|  | **T3** | ***2.56±0.98*** | 2.56±0.98 | 2.56±0.98 |  |
|  | **T4** | ***2.38±1.29*** | 2.38±1.29 | 2.38±1.29 | 2.38±1.29 |
|  |  | **T0** | **T1** | **T2** | **T3** |

p>0.5; *p<0.05*; ***p<0.01***; **p<0.001**

Table S5: Mean and standard deviation of normalized replicates within each chemical for the 6 h batch. Results of repeated measures ANOVA for the 6h batch—significance of the effect of time noted.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Overall | **T1** | 0.28±0.06 |  |  |  |
|  | **T2** | −0.48±0.35 | **−0.48±0.35** |  |  |
|  | **T3** | −0.53±0.53 | **−0.53±0.53** | −0.53±0.53 |  |
|  | **T4** | **−0.83±0.13** | **−0.83±0.13** | *−0.83±0.13* | *−0.83±0.13* |
| NaClO 12.5% | **T1** | −0.59±0.45 |  |  |  |
|  | **T2** | ***−1.65±0.45*** | **−1.65±0.45** |  |  |
|  | **T3** | **−1.94±0.33** | **−1.94±0.33** | *−1.94±0.33* |  |
|  | **T4** | **−2.18±0.44** | **−2.18±0.44** | −2.18±0.44 | −2.18±0.44 |
| NaClO 2.5% | **T1** | 0.92±0.42 |  |  |  |
|  | **T2** | 0.29±0.66 | *0.29±0.66* |  |  |
|  | **T3** | −0.11±0.62 | **−0.11±0.62** | −0.11±0.62 |  |
|  | **T4** | 0.30±0.68 | **0.30±0.68** | **0.30±0.68** | ***0.30±0.68*** |
| H2O 6h | **T1** | 0.50±0.54 |  |  |  |
|  | **T2** | −0.67±1.13 | −0.67±1.13 |  |  |
|  | **T3** | 0.47±1.36 | 0.47±1.36 | 0.47±1.36 |  |
|  | **T4** | 0.30±0.68 | 0.30±0.68 | 0.30±0.68 | 0.30±0.68 |
|  |  | **T0** | **T1** | **T2** | **T3** |

p>0.5; *p<0.05*; ***p<0.01***; **p<0.001**

Table S6: Spearman Correlation coefficients between sequences analysed with the same chemicals.

|  |  |  |
| --- | --- | --- |
| Chemical Treatment | Sequences | Spearman Correlation |
| H2O2 33% | A vs D | 0.223 |
| A vs G | 0.549\* |
| D vs G | 0.517\* |
| NaClO 12.5% | B vs E | 0.512 |
| B vs H | 0.653\* |
| E vs H | 0.741\*\* |
| HNO3 50% | C vs F | 0.824\*\* |
| C vs I | 0.591\* |
| F vs I | 0.685\*\* |

\*\*Correlation significant at the 0.01 level \*Correlation significant at the 0.05 level

Table S7: Spearman Correlation coefficients between numbers (# cm¯³) and area (mm³ cm¯³) of fragments for the nine fossil sequences. Chemical treatment used to analyse each sequence also given.

|  |  |  |
| --- | --- | --- |
|  | Treatment | Spearman Correlation |
| SeqA | H2O2 33% | 0.898\*\* |
| SeqB | NaClO 12.5% | 0.897\*\* |
| SeqC | HNO3 50% | 0.937\*\* |
| SeqD | H2O2 33% | 0.951\*\* |
| SeqE | NaClO 12.5% | 0.833\*\* |
| SeqF | HNO3 50% | 0.923\*\* |
| SeqG | H2O2 33% | 0.936\*\* |
| SeqH | NaClO 12.5% | 0.916\*\* |
| SeqI | HNO3 50% | 0.962\*\* |

\*\*Correlation significant at the 0.01 level

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