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Mordant Selection in Natural Dyeing of Cotton with *Cinnamomum verum* Bark Extract: Colour Strength against Rubbing Fastness

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***Abstract* - Natural dyes are applied with mordants, and the metal salts commonly used for the purpose differ substantially in environmental burden: copper salts in particular are toxic to aquatic life and are discharged in dyehouse effluent. This study compares alum and copper sulphate as pre-mordants for Cinnamomum verum bark extract on 100% bleached cotton, across aqueous and methanolic extraction systems at three dyebath liquor ratios, evaluating both colour strength by the Kubelka-Munk function and rubbing fastness by ISO 105-X12. Copper sulphate gave the higher colour strength in every comparison, reaching a peak K/S of 7.6 against 3.5 to 4.5 for alum. Rubbing fastness ran in the opposite direction: alum-mordanted fabrics reached grade 4 dry and grade 3 wet, while several copper-mordanted fabrics fell to grade 1/2 wet. The deeper shades obtained with copper sulphate were thus accompanied by poorer resistance to rubbing. Depth and mordant identity vary together in this design, so the poorer rubbing fastness is not attributed to copper as such, but the practical consequence stands: the greater depth available from copper sulphate cannot be had without the accompanying loss of rubbing fastness, and copper is the more environmentally burdensome of the two metals. Where depth of shade is not the governing requirement, alum is preferable on both counts. Substituting a plant colourant for a synthetic one while retaining a metal mordant moves the environmental burden rather than removing it.**

***Keywords* - Cinnamomum verum; natural dyeing; mordant; colour strength; rubbing fastness**

I. INTRODUCTION

The colouration of textiles accounts for a substantial share of the water consumed and the effluent discharged by the textile industry. Synthetic dyes dominate the sector because they offer reproducible shades, a wide range of colours and generally good fastness, but their manufacture and application release coloured and chemically loaded wastewater, and a proportion of the dye applied is never fixed to the fibre and leaves in the effluent. Concern over this burden has renewed interest in colourants of plant origin, which are biodegradable and derived from renewable sources.

Natural dyeing, however, is not automatically harmless to the environment, and the dye is only one component of the process. Most plant colourants have limited substantivity for cellulose and must be applied with a mordant, a compound that forms a bridge between the colourant and the fibre. The mordant is frequently a metal salt, and the metals in common use differ considerably in their environmental behaviour. The environmental character of a natural dyeing process is therefore determined as much by the mordant as by the colourant, a point that is often passed over when natural dyeing is presented as a sustainable alternative.

Copper salts illustrate the difficulty. Copper sulphate is among the most effective mordants for tannin-bearing plant dyes and is routinely reported to give the highest colour strength of the metallic mordants in common use [2], [3]. It is also a recognised aquatic toxicant, and residual copper leaves the dyehouse in spent mordant liquor and in the exhausted dyebath, where it is not degraded but persists in the receiving water and in sediment [1], [2]. Replacing a synthetic colourant with a plant colourant while retaining a copper mordant moves the environmental burden rather than removing it, and may in some respects worsen it.

Sri Lanka is among the world's principal producers of Cinnamomum verum, and its bark contains tannins, flavonoids and other polyphenolic compounds capable of colouring textile fibres [4], [5]. Cinnamon bark extract has been applied to protein substrates in several studies. Adeel and co-workers dyed bio-mordanted silk under microwave assistance [6] and bio-mordanted wool [7], obtaining yellow shades in both cases; Kaya and Sen dyed a cotton/wool blend and reported mechanical and fastness performance [8]; and a further study on wool compared metallic with bio-mordants and reported copper sulphate to give the highest colour strength of those examined [6].

Work on cellulosic substrates alone is less common, and cotton presents a different problem from wool and silk. Cellulose offers hydroxyl groups rather than the amino and carboxyl groups of protein fibres, which co-ordinate readily with metal ions and with polyphenols, so results obtained on wool and silk do not transfer directly to cotton. Where cotton has been studied, attention has generally been concentrated on colour yield. Yet colour yield is not the property that determines whether a dyed fabric is serviceable: a deep shade that transfers to adjacent surfaces under abrasion is of limited practical value, and durability is the

property on which natural dyeings are most often found wanting.

This study accordingly compares alum and copper sulphate as pre-mordants for cinnamon bark extract on cotton, evaluating the two mordants on colour strength and rubbing fastness together rather than on colour yield alone, and considers the outcome alongside the environmental burden of the two metals as reported in the literature. The question addressed is whether the colour advantage conferred by copper sulphate is accompanied by a corresponding advantage in durability, or whether it is obtained at a cost that is not visible when colour strength is measured in isolation.

II. Background

A. Colouring Constituents of Cinnamon Bark

Cinnamon belongs to the Lauraceae and is available commercially in two principal forms [4], Ceylon cinnamon (Cinnamomum verum) and cassia, which differ markedly in coumarin content. The colour of the bark is attributed to several classes of compound acting together. Cinnamaldehyde contributes yellowish to brown shades and is responsible for the characteristic aroma; tannins contribute brown colouration and are themselves capable of acting as natural mordanting agents; and flavonoids and other polyphenols produce yellow to reddish-brown shades whose expression depends on pH. These constituents absorb strongly at short wavelengths and reflect in the longer-wavelength region, which accounts for the brownish-yellow appearance characteristic of cinnamon-derived dyeings.

Tannins matter particularly here: their adjacent hydroxyl groups chelate metal ions, so the colour obtained is a property of the extract and the metal together, and the same extract applied with different metals yields visibly different shades.

B. Cotton as a Substrate

Cotton fibre consists principally of alpha-cellulose, whose glucose units each carry three hydroxyl groups; dye uptake occurs predominantly in the less ordered amorphous regions of the polymer. Because cellulose presents only hydroxyl groups, it lacks the amino and carboxyl sites through which protein fibres bind metal-dye complexes, and mordanting is correspondingly more important on cotton than on wool or silk.

C. Mordanting Methods and Mordant Selection

Three mordanting sequences are in general use: pre-mordanting, in which the mordant is applied to the fabric before dyeing; meta-mordanting, in which mordant and dye are applied together; and post-mordanting, in which the mordant is applied after dyeing. Pre-mordanting has been reported to give the higher colour strength for tannin-bearing dyes, while post-mordanting has been associated with better washing and light fastness [11]. Pre-mordanting was adopted throughout the present work so that the mordant comparison is made under a single, consistent sequence.

D. Metal Mordants and Dyehouse Effluent

Mordanting is an exhaust process, and exhaustion is never complete. Part of the metal salt remains in the spent mordant liquor and more is removed during rinsing and dyeing; residual metal contents are seldom reported, but have been estimated to exceed environmental release limits even at the lowest salt concentrations in common use [2]. The metal that does not fix to the fibre leaves the dyehouse in the effluent.

The two metals differ at this point. Aluminium salts have been reported to carry the lower pollution load of the mordants in common use [12]. Copper is toxic to aquatic organisms at low concentration, is not degraded, and accumulates in sediment and in exposed organisms. Its removal from effluent is feasible by precipitation, adsorption or membrane processes [13], [14], but each needs equipment, expertise and monitoring that small and medium-scale dyers may not have.

The difference between the two metals follows from the way each binds the colourant. Tannins and related polyphenols chelate metal ions through their ortho-dihydroxyphenolic groups, and transition metals, which have partly filled d orbitals available for bonding, form stronger and more stable chelates with such ligands than hard trivalent cations of comparable charge [10]. Copper(II) is a transition metal and aluminium(III) is not, which is the usual explanation for the higher colour yields obtained with copper salts on tannin-bearing plant dyes; mordanted fabrics have been reported to reach roughly twice the colour strength of unmordanted ones, with the metal identity governing how much of that gain is realised [3].

Colour strength is expressed here by the Kubelka-Munk function, K/S, calculated from reflectance. For an opaque substrate K/S rises with the concentration of colourant present in the material, so a higher K/S indicates that more colourant has been taken up, and comparisons of K/S between samples are comparisons of depth of shade rather than of hue [11].

Resistance to rubbing is governed by a different quantity. The crockmeter test measures colourant transferred mechanically from the fabric surface to a rubbing cloth, so it responds to colourant that is present but not firmly held, rather than to the chemically fixed fraction that determines washing and light fastness. Deeper dyeings are for this reason widely observed to give poorer rubbing grades across dye classes, since more colourant is present at and near the surface. Colour depth and rubbing fastness are therefore not expected to improve together, and a mordant that raises the one may not raise the other [2].

Alum and copper sulphate were selected because they span the two ends of the practical range of the common metallic mordants. Alum has been reported to give good colour depth together with acceptable fastness [7], while copper sulphate has been reported to give the highest colour strength of the mordants examined in comparative studies [2], [3]. They also differ in environmental profile, which is the second axis on which this paper compares them.

III. Materials and Methods

A. Materials

The substrate was 100% bleached cotton fabric, verified as cotton before use. Ceylon cinnamon bark was harvested at maturity, indicated by the change of stem colour from green to brown, dried in shade without exposure to direct sunlight, ground to a fine powder and stored until extraction. Potassium aluminium sulphate (alum) and copper sulphate were used as mordants, distilled water and methanol as extraction media, and sodium chloride as levelling agent. Methanol replaced the ethanol used in previously reported work because of restrictions on the supply of ethanol and its cost.

B. Pre-mordanting

Pre-mordanting was applied before dyeing under conditions identical for both mordants, given in Table I. After mordanting, samples were dried for 24 h without contact with water and shielded from direct sunlight and other light sources. Applying both mordants under identical conditions is what permits the comparison reported here to be attributed to the identity of the metal rather than to the treatment.

Table I. Pre-mordanting Conditions, Identical for Both Mordants

Parameter	Condition
Mordant concentration (owf)	10%
Material-to-liquor ratio	1:25
Temperature	100 °C
Time	60 min
Drying	24 h, dark, dry

C. Dye Extraction

Two extraction systems were used, aqueous and methanolic, under the conditions given in Table II. Both extractions were carried out in a closed system to limit oxidation by atmospheric oxygen, and the methanolic extraction used a double-boiler arrangement because of the flammability of methanol.

Table II. Extraction Conditions for the Two Systems

Parameter	Aqueous	Methanolic
Medium	Distilled water	Methanol
Temperature	100 °C	65 °C
Bark-to-liquor ratio	1:25	1:10
Time	45 min	60 min

The two systems differ in temperature, bark-to-liquor ratio and duration simultaneously, each parameter having been set to suit the medium concerned; the boiling point of methanol, 64.7 °C, prevents extraction at the temperature used for the aqueous system. The two are therefore compared here as whole systems and not as a test of solvent effect. Because the comparison reported in this paper is between mordants, and each mordant appears within both extraction systems under otherwise identical conditions, this limitation does not affect the conclusion drawn.

D. Dyeing

Dyeing was conducted in a closed system. The methanolic extract is a solution of the extracted colourant in methanol, and the dyebath prepared from it comprised 80% of that extract and 20% distilled water by volume, so that the bath was predominantly methanol. A dyeing temperature of 80 °C was used for the aqueous extract; for the methanolic extract this was reduced to 65 °C after complete evaporation of the dyebath was observed at the higher temperature, which lies above the boiling point of methanol. Conditions are given in Table III.

Table III. Dyeing Conditions

Parameter	Aqueous	Methanolic
Dyebath	Aqueous extract as prepared	Methanolic extract 80%, water 20% (v/v)
Temperature	80 °C	65 °C
Time	45 min	45 min
Liquor ratios	1:20, 1:25, 1:30	1:20, 1:25, 1:30
Levelling agent	NaCl, 1 g/20 mL	NaCl, 1 g/20 mL

Samples are identified by a three-part code: the first character denotes the extraction medium (W, distilled water; M, methanol), the second the mordant (A, alum; C, copper sulphate) and the numeral the dyebath liquor ratio. WC20 therefore denotes the sample extracted in water, mordanted with copper sulphate and dyed at a 1:20 liquor ratio. Twelve treatment combinations result.

E. Evaluation

Colour strength was determined by reflectance spectrophotometry and expressed as the Kubelka-Munk function K/S over the range 360-700 nm. Rubbing fastness was determined according to ISO 105-X12 [15] using a crockmeter, with dry and wet white cotton rubbing cloths, the wet cloth carried to a moisture content of 95-100%, a force of 9 N applied over an area of 3 cm × 3 cm and 10 rubbing cycles. Staining of the rubbing cloth was assessed against the grey scale under a D65 standard source.

IV. Results

A. Colour Strength

All twelve samples produced K/S spectra of similar form across 360-700 nm, the maximum falling between 360 and 370 nm and the values decreasing progressively towards longer wavelengths. The maximum therefore lies at the short-wavelength limit of the measured range, so the values discussed below are peak values within the range measured rather than a maximum located within the visible region. Because the object of this paper is the comparison between mordants, colour strength is reported here by mordant and extraction system rather than sample by sample; the ranges are given in Table IV.

Table IV. Peak Colour Strength by Mordant and Extraction System

Mordant	Aqueous	Methanolic	Overall
Copper sulphate	5.0-7.6	6.0-7.2	5.0-7.6
Alum	3.5-4.5	4.1-4.2	3.5-4.5
Highest sample	WC20 (7.6)	MC25 (7.2)	WC20 (7.6)

Copper sulphate gave the higher colour strength in every comparison. The copper-mordanted samples ranged from 5.0 to 7.6 and the alum-mordanted samples from 3.5 to 4.5, an advantage of approximately twofold that is consistent across both extraction systems and all three liquor ratios. Copper ions are capable of forming co-ordination complexes with the tannins and polyphenols of the extract, and such complexes have been reported to raise colour yield on textile substrates [3], [10]; the measurements reported here establish the difference in colour strength but do not by themselves identify the mechanism, which would require spectroscopic evidence of dye-fibre and dye-metal interaction.

The effect of dyebath liquor ratio was not consistent between the two extraction systems. In the aqueous system colour strength fell as the liquor ratio rose, the 1:20 ratio giving the highest value; in the methanolic system the 1:25 ratio gave the highest, with 1:20 and 1:30 lower. The reason has not been established here. The effect is smaller than that of mordant and inconsistent in direction, so it does not affect the comparison reported.

B. Rubbing Fastness

Rubbing fastness, assessed as staining of the rubbing cloth against the grey scale, is given in Table V. The ranking is substantially the reverse of the colour strength ranking. The best performers were all alum-mordanted: WA25 reached grade 4 dry, and WA30, MA20 and MA25 grade 3/4 dry, with WA25, WA30 and MA20 all reaching grade 3 wet. The poorest wet grades, at 1/2, were recorded by WC20, WC25, WC30 and MC30, every one of them copper-mordanted. WC20, which gave the highest colour strength of the entire set at a peak K/S of 7.6, was among the poorest of the set for wet rubbing.

TABLE V. RUBBING FASTNESS (ISO 105-X12), GREY SCALE GRADES

Sample	Mordant	Dry	Wet
WA25	Alum	4	3
WA30	Alum	3/4	3
MA20	Alum	3/4	3
MA25	Alum	3/4	2/3
MA30	Alum	3	2/3
MC20	$CuSO_4$	3	2/3
WA20	Alum	3	2
MC25	$CuSO_4$	2	2
WC20	$CuSO_4$	2	1/2
WC25	$CuSO_4$	2	1/2
WC30	$CuSO_4$	2	1/2
MC30	$CuSO_4$	1/2	1/2

Considered by mordant rather than by individual sample, the alum-mordanted set averaged appreciably higher grades in both dry and wet rubbing, and contained every one of the best-performing treatments. All four of the poorest wet-rubbing treatments were copper-mordanted. The separation between the two sets is thus visible at group level as well as sample by sample. The stained rubbing cloths for all twelve treatments are shown in Fig. 1. The copper-mordanted set is also the deeper-dyed set, so the two vary together; this is taken up in sections V and VI.

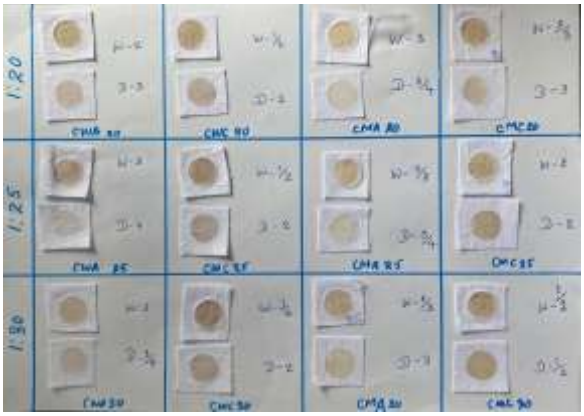


Fig. 1. Staining of the dry (D) and wet (W) rubbing cloths for all twelve treatments. Columns, left to right: aqueous extraction with alum (CWA) and with copper sulphate (CWC), then methanolic extraction with alum (CMA) and with copper sulphate (CMC). Rows are the dyebath liquor ratios 1:20, 1:25 and 1:30. Labels on the photograph carry a leading C for cinnamon, so CWA20 corresponds to WA20 in the text.

V. DISCUSSION

A. The Inversion Between Depth and Durability

The two properties measured run in opposite directions: the samples of greater colour strength were, with few exceptions, the samples of poorer rubbing fastness. Two readings of this are possible, and the present design cannot distinguish between them.

The first is specific to the metal. The copper-tannin complex responsible for the greater colour depth may not be correspondingly well fixed to the cellulose, so that a larger proportion of the colourant is present in a form available to transfer under mechanical action. Deposition of a poorly bound metal-tannin complex on and near the fibre surface would raise measured colour strength while contributing little to rubbing fastness.

The second is a general property of depth of shade. Rubbing fastness is a mechanical test, measuring colourant transferred from the fabric surface, and deeper dyeings are widely observed to give poorer rubbing grades across dye classes, because more colourant is present at and near that surface. On this reading the poorer performance of the copper-mordanted samples follows from their greater depth rather than from the identity of the metal.

Because copper sulphate produced the deeper shade in every pair examined, depth and mordant identity vary together throughout the set, and the two explanations cannot be separated from these measurements. Neither is asserted here. It is relevant that mordants are reported to improve washing and light fastness more consistently than rubbing fastness, and that no strong correlation has been found between mordant-induced changes in colour depth and improvements in fastness [2]; the present observation is therefore consistent with that unevenness rather than at odds with the function of a mordant.

The practical consequence holds under either reading, and that is what makes the observation useful: whether the poorer rubbing fastness follows from the metal or from the depth of shade it produces, the two

cannot be had apart, since within this dye system the greater colour depth available from copper sulphate is not obtainable without the accompanying loss of rubbing fastness. If colour depth were the only criterion, copper sulphate would be selected without hesitation, and this is the basis on which it is usually recommended in comparative studies of mordants. Once durability is also considered, the position reverses: the alum system delivers a fabric that resists rubbing substantially better, at a shade approximately half as deep.

B. Comparison with Reported Work on Protein Fibres

The colour strength ordering reported here agrees with what has been found on wool and silk: copper sulphate outperforms alum on tannin-bearing plant dyes across substrates [2], [3]. That agreement is worth noting, because it indicates the ordering is a property of the metal-tannin chemistry rather than of the particular substrate.

The durability result, however, does not follow the protein-fibre literature, where copper-mordanted wool has been reported to give fastness grades in the good to very good range [11]. Two differences may account for this. Wool and silk present amino and carboxyl groups capable of co-ordinating the metal directly, so the metal-dye complex is anchored to the fibre through the fibre's own functional groups; cellulose offers only hydroxyl groups and no comparable anchoring. Second, the studies reporting good fastness on wool have in several cases used post-mordanting or bio-mordants rather than metallic pre-mordanting, and the sequence is known to affect fastness independently of the mordant [6].

C. Environmental Implications of Mordant Choice

The environmental dimension reinforces rather than complicates this reading. Copper is an aquatic toxicant, and residual copper is discharged in spent mordant liquor and in the exhausted dyebath, where it persists rather than degrading [2], [13]. Aluminium salts have been reported to carry the lower pollution load of the metal mordants in common use [12]. A process description that presents cinnamon dyeing as environmentally preferable to synthetic dyeing, while specifying a copper mordant, is therefore incomplete, and the comparison it invites is not the one that matters: the relevant question is not whether the colourant is of plant origin but what leaves the dyehouse.

In the present dye system the mordant carrying the lower environmental burden is also the one producing the more durable fabric. The two considerations that might have been expected to conflict, environmental performance and serviceability, in fact point the same way, and the case for copper sulphate rests on colour depth alone. This is a more favourable position than is often encountered in sustainable processing, where improved environmental performance is typically obtained at a cost in product quality.

D. Implications for Practice

Mordant selection for cinnamon bark dye on cotton should accordingly be treated as a decision with three considerations rather than one. For applications subject to abrasion in wear, alum at a 1:25 or 1:30 dyebath liquor ratio in aqueous extraction is preferable on both durability and environmental grounds, despite its lower colour strength. Copper sulphate remains defensible where depth of shade governs, abrasion is limited, and copper-bearing effluent can be treated. Reporting either mordant as superior without reference to the other property misrepresents the choice.

TABLE VI. INDICATIVE MORDANT SELECTION GUIDANCE

End use	Governing property	Indicated mordant
Apparel, abrasion in wear	Rubbing fastness	Alum
Furnishing, low abrasion	Depth of shade	Copper sulphate
Deep shade, treated effluent	Depth of shade	Copper sulphate
Deep shade, untreated effluent	Effluent metal load	Alum
Small-scale, no treatment	Effluent metal load	Alum

Table VI sets out the indication in summary form. It is offered as guidance derived from the two properties measured and from the environmental characteristics cited, not as a validated specification, and it rests on the association between depth and rubbing fastness reported here rather than on an established causal effect of the metal; the fastness requirement for any particular end use should be established against the relevant product standard.

VI. LIMITATIONS

Four limitations bear on the interpretation of these results. First, the values reported are single determinations, and no replicate measurements or statistical analysis were carried out; the smaller differences between individual samples should therefore be regarded as indicative rather than established. The comparison between mordants, being approximately twofold and consistent across all six pairs in both extraction systems, is considerably more secure than comparisons between adjacent liquor ratios.

Second, colour strength was characterised by the Kubelka-Munk function alone, which expresses depth of colour and not hue. Colourimetric coordinates in the CIELAB system would be required to characterise the shades objectively, and any description of the shades obtained rests on visual assessment.

Third, depth of shade and mordant identity are confounded. Copper sulphate produced the deeper shade in every pair examined, so the samples of greater colour strength are also the copper-mordanted samples, and the poorer rubbing fastness of that set cannot be attributed to the metal rather than to the depth of dyeing it produced. Separating the two would require samples of matched colour strength prepared with each mordant, which this design does not provide. No unmordanted control was included either, so the results compare two mordants with one another and say nothing about the effect of mordanting as against its absence.

Fourth, no effluent analysis or life-cycle assessment was undertaken in this work. The environmental burden attributed to the two mordants is taken from the published literature and is not a measurement reported here. The paper compares two textile properties and reads the comparison against a cited environmental fact; it does not itself measure environmental performance.

VII. Conclusions

Alum and copper sulphate were compared as pre-mordants for Cinnamomum verum bark extract on 100% bleached cotton, across two extraction systems and three dyebath liquor ratios, and evaluated on colour strength and rubbing fastness.

Copper sulphate gave approximately twice the colour strength of alum, reaching a peak K/S of 7.6 against 3.5 to 4.5 for the alum-mordanted samples, and the advantage was consistent across both extraction systems. Rubbing fastness ran in the opposite direction: alum-mordanted fabrics reached grade 4 dry and grade 3 wet, while several copper-mordanted fabrics, including the sample of highest colour strength, fell to grade 1/2 wet.

Greater colour depth was therefore obtained together with poorer rubbing fastness, and from the mordant carrying the higher reported environmental burden. Because depth and mordant identity vary together in this design, the poorer rubbing fastness is not attributed to copper as such; what the results establish is that the two are not separable in practice, so that the additional depth available from copper sulphate is not obtainable without the accompanying loss of rubbing fastness. Selection between the two mordants is accordingly a trade-off rather than an optimisation, and for end uses in which maximum depth of shade is not required, alum is preferable on both durability and environmental grounds. More broadly, the result indicates that the environmental case for a natural dyeing process cannot be made from the origin of the colourant alone.

Further work should compare the two mordants at matched colour strength, which would separate the effect of the metal from the effect of depth of shade and settle the question left open here, and should include an unmordanted control. It should also establish whether the rubbing fastness of the copper-mordanted system can be improved by after-treatment or by a natural fixing agent; whether bio-mordants [16] can approach the colour yield of copper sulphate without its metal load; and whether the mechanism proposed here for the inversion between depth and durability is supported by spectroscopic and microscopic evidence. Measuring the shades in the CIELAB system, and repeating each measurement so that the differences can be tested statistically, would make the comparison more reliable.

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