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Development of New Powder Flux Lubrication Compound for Mild Steel Wire Drawing IV Okoro and S C Nwigbo

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ABSTRACT

Application of wax and other petroleum-based compounds as a wire drawing lubrication compound after a very long period is now discouraged due to its carcinogenic nature. There is need for alternative lubrication material. Hence, the development of petroleum-free lubrication material from an agro-waste as a base element with other additives was undertaken using simplex centroid mixture technique, the elements were characterized. Based on the investigated chemical characterization of the developed lubricant, increase in kernel fibre content increased the tensile strength, hardness and uniform wear of the drawn wires. The results showed that agro - waste could be effectively used as replacement for wax and other petroleum-based compounds in mild steel wire drawing lubricant manufacture. The blends and error analysis in this research was evaluated using design expert. Wire drawing operation was carried out using developed and commercial lubricants. In this experimental study, the results of hardiness and tensile strength tests ran with the wire drawn with produced lubricant are 488.5N and 515Mpa respectively which compares relatively well with the following results, 502.3N and 532Mpa of wire drawing lubricant imported into the Nigerian market on hardiness and tensile strength tests respectively.

Key words: Lubricant, Palm bunch Waste, palm kernel fibre, Mechanical Properties, Experimental Design (simplex centroid mixture technique), Material Characterization.

1. INTRODUCTION

Tribology is defined as the science and technology of interacting surfaces in motion and of related subject and practice (Arnell *et al.*, 1991; Halling, 1998). It has also been defined as the science and technology of friction, lubrication and wear (Szeri, 2002). The problems associated to it are not just confined to the machines we use; they have profound influences on many aspects of life, like the actions of animal joints. The cures for diseases such as arthritis already owes much to the tribological expertise (Halling, 1998). Tribology has over time studied friction and wear as major causes of failure in machines. (Halling, 2004).

Friction is the resistance to relative motion of two solid surfaces in contact and can course power lost, overheat moving parts and wear in machines, which can lead to a failure in the system (McGannon, 1989). Friction and wear cause loss of millions of dollars annually in lost production, lost time, lost materials and wasted energy. Lubricants can be used more efficiently to reduce this cost (Szeri, 2005).

Until this time, lubricants had been made primarily from animal fats and vegetable oils. In response to growing demands, particularly from the burgeoning Auto industry, lubricant manufacturers begin processing their petroleum-based oils to improve lubricant performance. It is now generally known that during application, petroleum releases hazardous gases, which can cause damages to human health, and this is not desirable, thus ideas were geared towards searches for more human friendly materials. Petroleum is pronounced carcinogenic with associated diseases that include lung cancer and other cancers. This work aimed at obtaining a suitable lubrication materials, taking cost, performance and environmental implication into consideration.

2.MATERIALS AND METHODS

2.1 Materials

The materials selected for this work are: palm bunch waste, palm kernel fibre, clay, black dye, commercial mild steel, while laboratory equipment used included; oven, conventional cloth sieve of 126 x 10^{-4} micron mesh size, pot, Whiteman micro filter paper of 125 cm, electric hot plate, hammer milling machine, stirrer, bowls, digital electronic weigh balance, hand gloves, eye google, nose mask, identic universal hardness testing machine (type 8187.5 LKV model B), universal tensile testing machine (TUE – C – 100), Microscope, hack saw , measuring tape, pedestal grinding machine, wire drawing die, hand drilling machine, and mechanical vice. All laboratory and workshop equipment used were assessed at Standard Organization of Nigeria Enugu (SON) and Scientific Equipment Development Institute Enugu (SEDI-E) situated in Enugu State, Nigeria.

2.2 Materials preparations

Palm bunch wastes were collected from oil palm fruit processing facility at Nsude, Udi Local Government Area Enugu State. The palm bunch wastes were dried under the sun for two days, and heated at the temperature of 105°C in an oven for 6 hours, to reduce the water content in the palm bunch waste. The Palm bunch wastes were burnt and the ashes were subjected to further crushing, and then sieved with conventional cloth sieve of mesh size 126 x 10^{-4} x 10^{-6} micron to obtain uniform particle size. 350g of the ash was placed in a pot with 4 liters of water added to it. The container was heated to a boiling point (100°C) for 1 hour using an electric stove , the content of the pot was allowed to cool for 2 hours before it was filtered using a conventional filtered cloth, and re-filtered with Whitman micro filter paper of 125cm to obtain a clearer extract (Överstam, 2006). The filtrate was poured into another pot, placed on an electric hot plate and allow to dry. A solid residue (alkali) was obtained (nchaisiakwu), which was further sun dried and crushed, the powder (palm fruit bunch ash) was characterized at Standard Organization of Nigeria, Enugu lab. The chemical composition of the alkali (Palm bunch waste powder) is presented in table 1.

Palm kernel fibre (370grams) was obtained from oil mill at Nsude, Udi local Government Area in Enugu State, Nigeria. The fibre was washed, to reduce oil and dirt. It was sun dried for 7 hours, grinded down into a powdered form using a hammer-milling machine.

Clay sample used (493grams) was acquired from Udi in Enugu state, Nigeria. After removing the contaminant, the samples was grounded to increase the surface area and was dried.

Dye

Potash (Palm bunch waste)

Clay

Palm Kernel fibre

Fig. 1: List of materials used in the lubrication production

2.3 Chemical Characterization of the Constituents

The flux candidates were characterized at **standard organization of Nigeria Enugu (Engineering lab),** Palm bunch waste (potash) contains 83.5 %Wt of Potassium, which gives the flux a high level of flow (viscosity) during wire drawing. Palm fruit fibre contain 86 %Wt. of Chlorine, which promotes the boundary condition of the lubricant. Clay presents 6 elements oxides, which is a very good hydrodynamic lubricant of the flux

Table 1 Chemical Characterization of palm fruit bunch

Parameter	%wt.
Carbon	0.03
Oxygen	7.34
Magnesium	1.69
Phosphorus	5.38
Sulphur	1.55
Chlorine	3.47
Potassium	83.5

Table 2 Chemical characterization of clay

Parameter	**%Wt.**
Potassium oxide	0.14
Silicon oxide	55.72
Aluminium oxide	6.86
Iron (11) oxide	0.21
Lead oxide	0.01

Table 3 Chemical characterization of palm fruit fibre

Parameter	%Wt.
Carbon	50.80
Hydrogen	5.62
Oxygen	35.61
Nitrogen	0.69
Chlorine	86 ppm

2.4 Development of Design Matrix

The experiment was designed using a design expert 10.3, utilizing scheffe simplex centroid mixture method. The limit values of palm bunch waste, palm kernel fibre, clay and black dye were keyed in the appropriate dialogue box of the design expert, and the possible blends were generated by the program or read from the file as shown in Table 5. There were twenty-one experimental runs consisting of eleven (11) feasible design points, five (5) space-filling points and five (5) additional centre points.

Since this is a multi-component mixture, limitations are being i mposed on the upper and lower limits of the mixture. The form of the constrained mixture is given as

$A + A_{2+}A_{3.....+An=1}$ 1

$X_i \leq A_i \leq Y_i$ $for\ i = 1,2,3,4, \ldots . n\ where$ Xi ≥ 0and Yi ≥ 1

Xi ≤ Ai ≤ 1 for I = 1, 2, 3, 4 ….n 2

Where

X_i is the lower limit

Y_i is the upper limit

A_i is the independent components

i = 1, 2, 3, 4 …n (the number of component)

Considering 4-component mixture

$Xi = Ai + Ai + Ai + Ai \geq 0$ 3

$Xi = 0.50 + 0.15 + 0.10 + 0.1 = 0.85 \geq 0$ 4

$Yi = A_{i+}A_{\ i} + A_{i\ +} A_{\ i\ \geq 1}$

$0.60 + 0.20 + 0.15 + 0.5 \geq 1 = 1.45 \geq 1$ 5

The limitation or constrain imposed on the upper and lower limits of the mixture in order to achieve optimal blends is that, their summation must be greater or equal to one and zero ($Yi \geq 1$ and $Xi \geq 0$), respectively

Table 4. Iteration limits of the constituents

S/n	Constituents	Lower limit (%Wt.)	Upper limit (%Wt.)
1	Potassium Soap	50.0	60.0
2	Palm kernel fibre	15.0	20.0
3	Clay	10.0	15.0
4	Black dye	1.0	5.0

Table 5 Composition of the new developed flux

Run Order	Potassium (% wt.)	Palm Fibre (% wt.)	Clay (% wt.)	Dye (% wt.)
1	51.250	15.625	10.625	3.500
19	50.000	15.000	10.000	1.000
9	55.000	15.000	10.000	3.000
20	52.500	16.250	11.250	2.000
14	50.000	16.666	11.666	2.333
5	50.000	15.000	10.000	1.000
2	51.250	15.625	10.625	1.500
21	25.500	16.250	11.250	2.000
11	53.333	16.666	11.666	1.000
10	50.000	15.000	10.000	1.000
8	53.333	16.666	11.666	1.000
7	50.000	17.500	12.500	3.000
18	55.000	17.500	15.000	1.000
4	56.250	15.625	10.000	1.500
12	50.000	15.000	12.500	3.000
3	51.250	18.125	10.625	1.500
6	55.000	15.000	12.500	1.000
16	60.000	15.000	10.000	1.000
13	53.333	15.000	11.666	2.333
17	50.000	20.000	13.125	1.000
15	52.500	16.250	11.250	2.000

Errors were generated during blending of the mixtures. The values were estimated using Design point graph (DP). Figure 2, shows the DP graph for the blend.

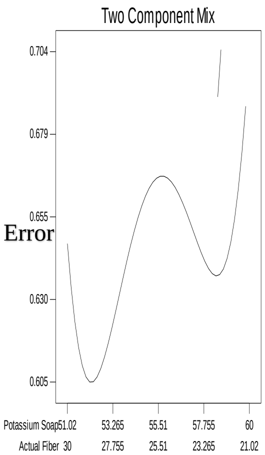

Fig 2. Design points graph

The mean FDP (fraction design point) curve is quite low and not steep. The minimum standard mean error is 0.630 and the maximum standard mean error is 0.704. These are quite good for the generated experiment runs in this study.

The tolerable components composition limits within the available design point were also evaluated by mixture triangle DP graph as shown in Fig. 3. It was discovered that the best blend (minimal error of design) occur at component variation of 50.737 to 56.912%wt potassium soap, 17.649 to 26.912%wt fibre, and 10.737 to 16.912%wt clay at a constant value of 2.351%wt dye. This can be observed in Fig.3, since, those ranges of component variation lies within the tolerable error contours limit.

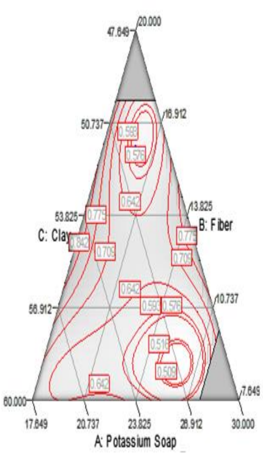

Fig 3: Error contours of DP Graph

2.5 Experiment

The conducted experiments processes were presented in subsequent subsection **2.8-2.10**

2.6 Powder Wire Drawing Lubricant Production

Wire drawing lubricants were produced from design matrix table values using palm bunch waste ash, palm kernel fibre, clay and black dye. For each mixture, the required amount of each element was calculated using equation 6.

$$A = 0.01/RO/Q/ \qquad 6$$

Where

A= quantity of constituent (g)

RO = run order value of the constituents (%wt.)

Q= quantity of the flux (g)

Fig. 4: Produced Lubricant

2.7Wire – drawing process.

Drawing operation was carried out using a wire-drawing die, with a working region of W-carbide. The produced and commercial lubricant were used during the drawing process, and their performances were evaluated. The reduction ratio (R %) was determined using equation (7)

$$R = Di - D0/Di \times 100 \qquad 7$$

7 Wire drawing process was carried out with different produced lubricants, to determine how the mechanical properties of the drawn wires were affected.

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Fig. 5: Drawn wire

2.8 Tensile test

The drawn wires were subjected to tensile teste machine with model number TUE-C-100, range of 100kW and cross head speed of 5 mm/sec at Standard Organization of Nigeria (Engineering lab) in Enugu State. The samples were at first fix in the chucks of the testing machine, the machine was zeroed, balanced, calibrated and the conditions of the test were selected. Then, load was applied on the specimen until it breaks. The tensile strength was determined from the stress-strain diagrams.

2.9 Hardness test

The hardness of the drawn wires measurement was done by universal hardness tester, utilizing ball indenter at an applied load of 10 kg. The aim of this test was to determine how plastic deformation actually affected the drawn wire specimens; this was done using ASTM E18-17e1, Standard Test Methods for Rockwell Hardness of Metallic Materials. The values obtained for different drawn wires were presented in Table 6.

2.10 The metallographic analysis

Metallographic examination was done to observe the microstructure of the drawn wires and the control sample using Optical Microscopy. A solution of 90% ethanol and 10% nitric acid was used for etching. The results are presented in Figure 8 - 11.

3. RESULTS AND DISCUSSIONS

3.1 Tensile Test

Figure 6 shows the results of the tensile test conducted on drawn wire samples, using produced and commercial lubricants under the same drawing conditions (Drawing speed and Drawing die). It can be physically observed that the pattern of stress – strain curves for all the drawn samples (drawn wires and control sample) are similar, but a wire sample drawn with produced lubricant typeA1(50)A2(20)A3(10)A4(1**)**, has relatively higher load at yield (380.26Mpa), elongation at yield (3.51mm),yield stress (423Mpa) and load at break point (340.16Mpa), compared to that of control sample with load at yield (374.92Mpa), elongation at yield (3.23mm), yield stress (385Mpa), and load at break (510.29Mpa). On the other hand, control sample has relatively higher elongation at peak (19.1mm),but lower elongation at break(20.3mm) compare to the wire drawn with lubricant type **A1(50)A2(17.5)A3(12.5)A4(3),**with elongation at peak (17.73mm) and elongation at break (21.2mm).it can be deduced that the wire drawn with the produced lubricant type **A1(50)A2(17.5)A3(12.5)A4(3),**with mixture compositions of potassium soap 50%w, palm kernel fibre 17.5%w, clay 12.5%w, and dye 3%w,produced more ductile wire compared to control sample. It also gave rise to adequate elongation for the intended application. By comparing the load obtained, it is observed that the sample drawn with developed lubricant type **A1(50)A2(17.5)A3(12.5)A4(3),** is capable of absorbing more load (532Mpa), before failure compare to those wire samples drawn with lubricant types , A1(50)A2(20)A3(10)A4(1), A1(55)A2(17.5)A3(15)A4(1) and A1(51)A2(18)A3(10.6)A4(1.5), meanwhile the difference in the tensile strength of the wires drawn with the four produced lubricants must have been due to variation in the process parameters (variation of the constituents during the blending of the mixture). It could be further concluded that wire drawn with the produced lubricant type A1(50)A2(20)A3(10)A4(1**)**, is more ductile than the control sample, because ductile materials exhibits large yielding before failure.

In all the drawn wire samples, failure did not occur at the point of maximum loads rather, the failure occur at a breaking load point. This shows that, at a maximum load, there were more elongation before the samples failed. It can also be agreed that, the failure of the drawn specimens happened with a maximum plastic deformation, which can be concluded that, the drawn wire materials were still ductile after undergoing `wire drawing process.

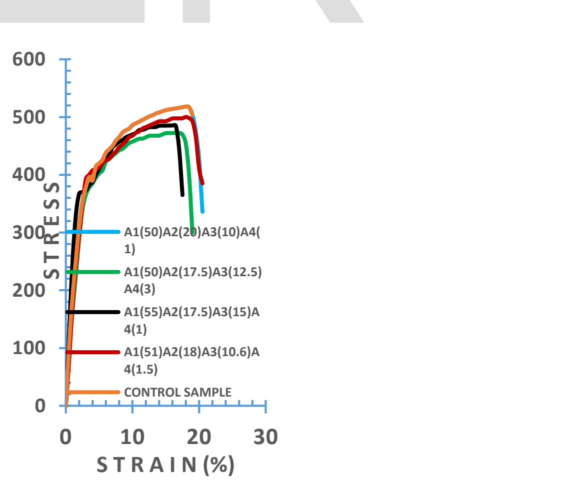

Fig. 6: Variation of stress-strain of tensile test conducted on the specimen drawn with produced lubricant types and control sample

3.2 Hardness Test

Figure 7 presents the hardness value graph of the drawn and control sample wires versus produced lubrication types and commercial lubricant. The hardness values of the wires drawn with the developed lubricants ranges from 273.7N to 488.5N and 502.3N for the wire drawn with commercial lubricant. Wire drawn with lubricant type A1(50)A2(20)A3(10)A4(1),with hardness of 488.5N and mixture compositions of Potassium Soap 50%W, Palm Kernel Fibre 20%w,Clay 10%w,dye 1%w, gave a higher hardness value of all the wires drawn with developed lubricant types. This was because of certain desirable properties in the blend (higher fibre content (20%), which is a boundary lubrication agent and a moderate content of clay (10%) that supports hydrodynamic lubrication condition). These desirable properties, reduces non-uniform distribution of temperature, strain and mechanical properties that could have imposed a negative effect on the hardness of the drawn wires. However, the wire drawn with commercial lubricant has higher result on hardness than the wires drawn with the produced lubricant, but the produced lubricants actually compete very well with the wire drawn with commercial lubricant. The little different between the two drawn wire samples (wire drawn with the produced and commercial lubricants), could be tagged to the materials and ideal methods adopted during the production of commercial lubricant. The ideal methods were believed to have reduced the drawing temperature of the wire drawn with commercial lubricant, which could have giving rise to higher strain hardness. This resulted to the slight higher hardness of the material drawn with commercial lubricant than the produced lubricant.

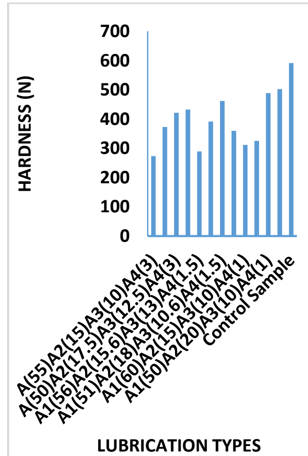

Fig. 7: Hardness test graph of the drawn wires

Table 6: Determined mechanical properties.

Prepared flux types/control sample	Tensile(Mpa)	Hardness test value(N)
1	499	462.1
19	418	391.4
9	316	273.7
14	402	372.8
5	372.8	488.5
11	399	325.7
Control sample	528	591.5
Commercial lubricant	528	502.3

3.3The metallographic analysis

Figure 8, 9, 10 and 11, shows the microstructures of the wires drawn with the new developed lubrication compound and control sample.

Fig. 8 Microstructure of a mild steel Control sample before wire drawing.

Fig. 9 Microstructure of the drawn steel wire with lubricant type A1(50)A2(17.5)A3(12.5)A4(3)

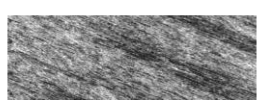

Fig. 10 the microstructure of the drawn steel wire with lubricant type A1(51)A2(18)A3(10.6)A4(1.5)

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Fig. 11 the microstructure of the drawn steel wire with lubricant type A1(50)A2(20)A3(10)A4(1).

Fig 8 shows the longitudinal metallographic photos of the disposition in the wire before drawing. It is clear to see from the picture that the typical sorbate grain organization and pearlite groups are small with equal axis, and the grains have no define directions along with the vertical or horizontal. In addition, there is little mesh precipitation (solid solution) of the ferrite in the grain boundary, with little grain refinement. Grain refinement is one of the most effective strengthening mechanism, improving mechanical properties without loss in ductility

Fig. 9 shows the microstructure photo of the deformed mild steel wire taken longitudinally after drawing process. It can be seen that the grains were refined in transverse direction, though the refinement were not so pronounced, due to low boundary lubrication condition during the drawing process, which resulted from low fibre content (17.5%) in the flux mixture type used, revealing coarse pearlite (black matrix) grain matrix in ferrite matrix (white matrix).etched×400 in the drawn wire. Boundary lubrication process maintains even - distribution of temperature, strain and mechanical properties during wire drawing excises, and evenly distributions of these parameters improves grain refinement during wire drawing. At higher temperature, when every other lubrication constituents (clay, palm bunch waste ash) failed, fibre will react with the metal surfaces (drawing die and wire surfaces) to form a soft firm (metallic chloride) to reduce metal-to-metal contact.

Figure 10 shows microstructure of the wire drawn with lubricant type **A1(51)A2(18)A3(10.6)A4(1.5),** taken longitudinally**.** More refined grains were revealed in this sample than the sample drawn with lubricant type **A1(50)A2(17.5)A3(12.5)A4(3).** The noticeable increased in the grain refinement at this drawn wire sample was believed to be as a result of parameter variations during blending of the lubricant types, (difference in fibre content between the lubricant types 18% and 17. 5% respectively, since increase in chlorine improves boundary lubrication condition.). The microstructure of the wire drawn with lubricant type **A1(51)A2(18)A3(10.6)A4(1.5)** also reveals a coarse structure of pearlite in ferrite matrix and a well-pronounced uniform wear than the wires drawn with other lubrication types.

The noticeable grain refinement and well- pronounced uniform wear in this drawn sample, could be attributed to the moderate fibre and clay contents of the lubricant type used (18% and 10.6% respectively), which promotes boundary and hydrodynamic lubrications respectively.

Fig. 11 shows the longitudinal microstructure photo of the drawn mild steel wire after drawing process using lubricant type **A1(50)A2(20)A3(10)A4(1)**, The grains were further refined and elongated in longitudinal section than other samples (drawn and control samples), revealing intermediate pearlite and elongated ferrite matrix. The further grains refinement in the drawn wire sample, can be attributed to the high content of palm kernel fibre (20%) in the drawing lubricant type used (20%w).

With the noted grain refinements and uniform wear observed in the microstructures of the wires drawn with the produced lubricant types, it is established that increase in palm kernel fibre (boundary lubrication agent/extreme pressure additive) and moderate clay (hydrodynamic lubrication agent) contents in a wire drawing lubricant, improves the mechanical properties of a drawn wire through grain refinement, by reducing drawing temperature.

A comparative study of drawing stress at dry and dynamic friction situation on mild steel wire of 6mm using T – Test, shows that the calculated T-value is greater than P-value, when the wire sample was drawn with the produced lubricant, and less than P-value when it was drawn dry (without lubricant). The calculated T- value should be large enough to give significant difference. T-value should also correspond to P-value. The hypothesis should be accepted if the calculated T – value is equal or greater than p –value and should be rejected if it is lesser.

Table 7: T- Test Statistical Analysis Values

Drawing stress (Mpa)	**With lubricant**	**Without lubricant**

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Mean Values	398.23	408.49
N	6	6
Standard deviation	23.016	26.475
Standard error	9.398	10.81
T - values	1.064	0.421
Degree of freedom	5	5

The examined drawing stress with lubricant has an adequate calculated T –value with adequate corresponding P – value, while the drawing stress without lubricant has no adequate P- value, rather it has a calculated T - value less than P – value. The mean difference = 10, degree of freedom of = 5. Looking this up in tables gives p = 0.544, the calculated T – values with and without lubricant are 1.064 and 0.421 respectively.

With the above comparative study, it can be clearly believed that there is a significant difference when the wires were drawn with and without lubricant. Therefore, there is a strong evidence that, on average, scheffer method is a very good tool for this design.

4.CONCLUSION AND RECOMMENDATIONS

4.1 Conclusion

The results showed that agro - waste has properties comparable to that needed for use as wire drawing lubricant material, to replace wax and other petroleum based compounds in the manufacture of wire drawing lubricant since it gave results, which are within the range for wire drawing lubrication manufacture. The results of the research show that agro - waste of 50 %wt of palm bunch waste, 20% wt of powder palm kernel fibre and 10% wt of clay has properties that can effectively replace wax and other petroleum-based compounds in wire drawing lubricant manufacture, since it gives comparable lubrication properties.

4.2 Recommendations

It is recommended that future research should supplement the palm bunch waste base lubricant with palm kernel shell, in order to draw both mild steel and stainless steel, but still reduce temperature and friction effectively.

Further study should consider and analyse the impurities content of the industrial waste, for they can affect the performance and efficient of the lubricant.

The federal government of Nigeria should encourage and improve the local production of lubricant using industrial waste, which is an act of recycling our waste and reducing environmental pollution.

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