Analysis of Potassium in Oil Palm Leaf and Rachis Samples: Is 'double calcination' required?

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Potassium deficiency symptoms in oil palm leaflets

SUMMARY:

The method of 'Double Calcination' digestion of plant material by using very corrosive acids such as hydrofluoric acid (*HF*), results in higher values of potassium (*K*) being detected than by 'Single Calcination' digestion. Single calcination is the method routinely used in many analytical laboratories (*such as AAR*), which the oil palm industry uses for nutrient analysis of palm tissues.

In spite of this 'more complete' digestion by the double calcination method, it is recommended that the oil palm industry continues to use 'single calcination' as the method of choice as it most likely reflects metabolically available K rather than total K.

BACKGROUND:

The results of analysis of chemical elements (*nutrients*) in plant material can be strongly influenced by other plant components; especially those that are resistant to chemical digestion. Silica is particularly relevant in this case as it is often quite high in palms (*and other monocotylendous species*). Silica can suppress the analytical results of some elements; of particular interest is potassium (*K*).

At OPRA's 2004 SAC meeting, this issue was raised and OPRA was asked to investigate the appropriateness of this technique (*viz 'double calcination'*) for analysis of K in oil palm tissues, based on a report by P. Quencez (*CIRAD, 2000*) commissioned by NBPOL.

This paper discusses the chemical analysis results presented in that report and the appropriateness of its recommendations for the PNG oil palm industry (*vis-à-vis 'double' vs 'single calcination'*). It does not discuss the fertiliser recommendations which form a substantial part of that report.

This discussion is based solely on the chemical analysis conducted by CIRAD on oil palm leaflet samples, and on the previous analysis by AAR of the same samples.

METHODS:

A subset of analyses (*15*) has been chosen for which ash, K, and silica have been determined on all samples by various methods. The results of these analyses have been further analysed to reveal relationships not evident in the original report. This data is available in Quencez (*2000; table 4*), but has been collated here in Table 1 for convenience.

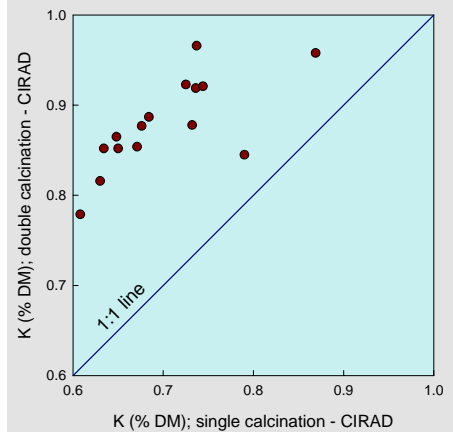Figure 1: Comparison of CIRAD's single and double calcination methods

Figure 2: Comparison of CIRAD's double calcination method with AAR's method

DISCUSSION:

The report clearly demonstrates that double calcination extracts more K than single calcination by the methods used at CIRAD (*Quencez, 2000, Graph 3; reproduce here for convenience as Figure 1*).

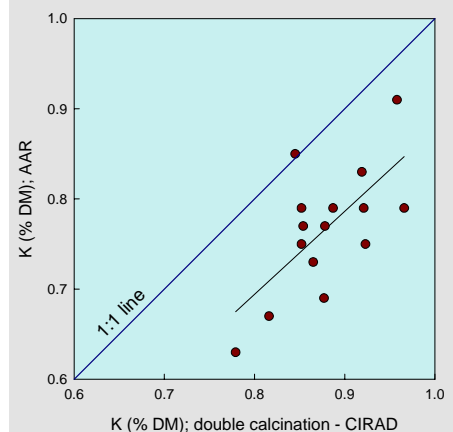However, what is not presented in the report, is the more pertinent question, "Is the CIRAD

Table 1: Subset of data from Quencez (2000) re-examined in this paper. (AAR: Applied Agricultural Resources Sdn. Bhd; dCIR, 'double calcination' CIRAD; sCIR, 'single calcination' CIRAD)

Sample ID	AAR Ash	AAR K	dCIR Ash	dCIR K	dCIR SiO$_2$	sCIR K
			---------- % DM ----------			
BBR101	14.78	0.79	15.55	0.887	11.10	0.684
BBR110	15.67	0.79	16.85	0.852	12.41	0.634
KBG206	14.80	0.83	10.10	0.919	10.73	0.736
KBG215	15.31	0.77	16.29	0.854	12.10	0.671
TGL401	15.09	0.69	15.52	0.877	11.37	0.676
TGL407	16.53	0.75	16.74	0.852	12.40	0.650
NVR502	15.08	0.67	15.48	0.816	11.35	0.630
NVR506	15.92	0.63	16.91	0.779	12.75	0.608
NVR508	15.63	0.79	16.49	0.921	11.84	0.744
MLL612	14.19	0.75	14.48	0.923	10.22	0.725
MLL616	13.25	0.85	13.59	0.845	8.80	0.790
HLA801	13.95	0.73	14.49	0.865	10.85	0.648
HLA805	13.45	0.77	14.20	0.878	8.97	0.732
NMD901	13.15	0.79	13.87	0.966	9.48	0.737
GRU1002	11.46	0.91	11.75	0.958	6.79	0.869

method of double calcination better at estimating leaf K levels than the currently-used AAR method?"

Table 2: Comparison of actual double calcination values for K with predicted double calcination values for K based on single calcination values for K and silica content.

Sample ID	dCIR K	dCIR SiO$_2$	sCIR K	Predicted dCIR* K
	---------------------------- % DM ----------------------------			
BBR101	0.887	11.10	0.684	0.870
BBR110	0.852	12.41	0.634	0.844
KBG206	0.919	10.73	0.736	0.901
KBG215	0.854	12.10	0.671	0.867
TGL401	0.877	11.37	0.676	0.866
TGL407	0.852	12.40	0.650	0.855
NVR502	0.816	11.35	0.630	0.836
NVR506	0.779	12.75	0.608	0.829
NVR508	0.921	11.84	0.744	0.913
MLL612	0.923	10.22	0.725	0.891
MLL616	0.845	8.80	0.790	0.925
HLA801	0.865	10.85	0.648	0.845
HLA805	0.878	8.97	0.732	0.889
NMD901	0.966	9.48	0.737	0.895
GRU1002	0.958	6.79	0.869	0.965

* Predicted dCIR = 0.361 + 0.651 * sCIR + 0.0057 * SiO$_2$
p = 0.010

A direct comparison of the two methods shows that the regression is significant (*although not 'tight'*) and the slope close to 1 (*Figure 2*). However, the values for the AAR method are generally lower than those for the CIRAD double calcination method.

So which method should be used in the PNG oil palm industry? The two methods give related but different results.

Table 3: Comparison of actual double calcination values for K with predicted double calcination values for K based on AAR values for K and AAR values for ash.

Sample ID	AAR Ash	AAR K	dCIR K	Predicted dCIR* K
	---------------------------- % DM ----------------------------			
BBR101	14.78	0.79	0.887	0.886
BBR110	15.67	0.79	0.852	0.877
KBG206	14.80	0.83	0.919	0.900
KBG215	15.31	0.77	0.854	0.873
TGL401	15.09	0.69	0.877	0.846
TGL407	16.53	0.75	0.852	0.853
NVR502	15.08	0.67	0.816	0.839
NVR506	15.92	0.63	0.779	0.816
NVR508	15.63	0.79	0.921	0.877
MLL612	14.19	0.75	0.923	0.877
MLL616	13.25	0.85	0.845	0.924
HLA801	13.95	0.73	0.865	0.873
HLA805	13.45	0.77	0.878	0.892
NMD901	13.15	0.79	0.966	0.903
GRU1002	11.46	0.91	0.958	0.964

* Predicted dCIR = 0.752 + 0.364 * AAR-K - 0.0104 * AAR-ash
p = 0.017

What we need to consider here is not the efficiency of K extraction by the methods (*of which the CIRAD double calcination is far superior*), but the biological significance of the K that is extracted.

If we again compare the two CIRAD methods (*Figure 1*), we can ask "Why is the 'double' method more efficient than the 'single' method?"

The answer, as suggested in the report, relates to the high silica content of oil palm leaves. Their results (*report graph 3; bottom p.15*) show there is almost a 1:1 relationship between double and single calcination methods for their standard reference material (*T cirad*) which is low is silica (*2.5% DM*). By contrast, the high silica (*9–13% DM*) palm leaves do not show the same 1:1 relationship.

Indeed, multiple linear regression analysis shows that the K values from double calcination can be predicted from the K values from single calcination plus the silica content (*Table 2 and Figure 3*).

The interpretation, from an analytical chemist's perspective, would be that the difference in the K values between the double and single calcination methods, is due to the K 'locked up' in the silica.

If this interpretation is correct, that is, that some K is locked up in the silica, then we must ask "Does the double calcination method tell us what is biologically available or does it simply reflect an analytical chemist's desire to extract every bit of K that is there?"; it is most likely the latter.

For this reason, it is OPRA's recommendation, that the current method of analysing K, while admittedly does not account for all of the K, is a better indication of what K is available to the palm for metabolic processes such as vegetative growth and FFB production.

RECOMMENDATION:

OPRA recommends that the current analytical method for measuring K in palms leaves (*and probably also in rachis*) be continued. While they may not accurately measure total K, they probably give a result that is more relevant in a biological sense.

FOOTNOTE:

If estimates of total K are still required, a good approximation can be obtained from current analytical measurements.

There is s strong relationship between silica (*CIRAD double calcination*) and ash (*AAR*) (*Figure 4*).

There is also a reasonable relationship between AAR-K and dCIR K (*Figure 2*). Thus it may be possible to predict total K (*i.e. dCIR-K*) from values of AAR-K and AAR-Ash. Again, multiple linear regression analysis can be used (*Table 3, Figure 5*). Clearly, it would be possible to get a reasonable estimate the total K, as measured by the CIRAD double calcination method (*if that was desired*) based on values already obtained from AAR.

Figure 3: Comparison of actual double calcination values for K with predicted double calcination values for K based on single calcination values for K and silica content.
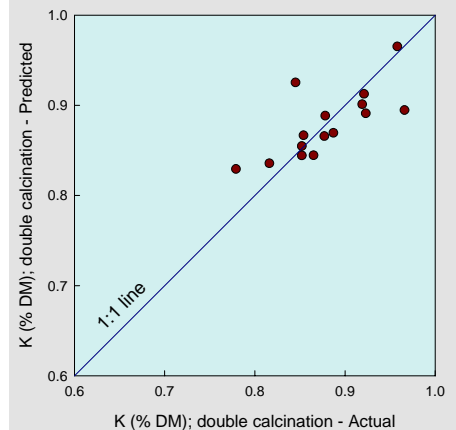

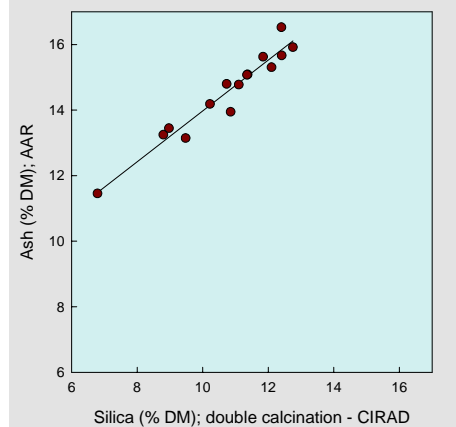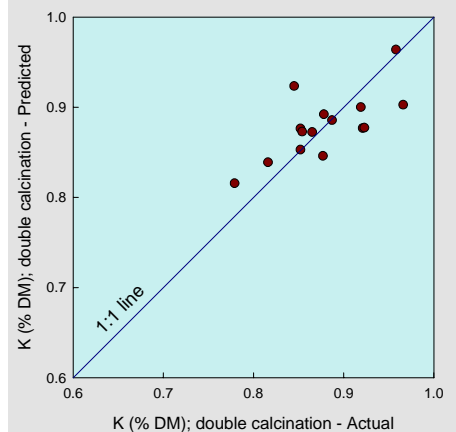Figure 4: Relationship between Ash (%DM) and silica (% DM) content.

Figure 5: Comparison of actual double calcination values for K with predicted double calcination values for K based on AAR values for K and