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The objective of this study was to determine possible correlations between some selected carbon and nitrogen assimilating enzymes and grain yield of maize. These enzymes were evaluated for possible use as biochemical markers for the development of new cultivars with improved nitrogen use efficiency. The double cross hybrid BR 201, and its two parental single cross hybrids BR 201F and BR 201-M, the reciprocal double cross hybrid BR 201-R, the quality protein variety BR 451, and the single cross hybrid 20x22 were planted under 4 nitrogen treatments; inoculation with *Azospirillum* and 10, 60, and 180 Kg.ha⁻¹ of nitrogen, replicated 4 times. The activity of phosphoenolpyruvate carboxylase (PEP-case), glutamine synthetase (GS), ferredoxin-dependent glutamate syntase (Fd-GOGAT), and the levels of ribulose-1,5-biphosphate carboxylase (RUBISCO) and soluble protein were determined at silking on the second leaf above the main ear. Grain yield was determined after black layer formation. Enzyme activity was calculated as International Units, expressed as $\mu\text{mols.g}^{-1}$ fresh weight.min⁻¹, and RUBISCO (ELISA) and soluble protein (Lowry assay) as mg.g⁻¹ fresh weight. *Azospirillum* inoculation did not influence any of the parameters measured, whereas the nitrogen levels strongly influenced them. The reciprocal BR 201-R yielded significantly less than BR 201. The single cross hybrids BR 201-F and 20x22 tended to be more productive than the other genotypes. A low, but highly significant correlation was found between yield (g.plant⁻¹) and the activity of PEP-case, GS, and Fd-GOGAT, and the levels of RUBISCO and soluble protein. Multiregression indicated that under low soil nitrogen levels, genotypes with increased contents of RUBISCO, soluble protein and PEP-case activity were more nitrogen use efficient.

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